

# VON KARMAN CENTER

SNAP-8 DIVISION

SNAP-8 MATERIALS REPORT FOR JANUARY-JUNE 1964

VOL. II - DEVELOPMENT OF COMPONENT MATERIALS

BY H. DEROW AND B. E. FARWELL

CONTRACT NAS 5-417

A REPORT TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION  
LEWIS RESEARCH CENTER

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A Report To

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SNAP-8 PROJECT OFFICE

H. O. SLONE, SNAP-8 PROJECT MANAGER

Report No. 2880

July 1964

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**AEROJET-GENERAL CORPORATION**

A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

## FOREWORD

Aerojet-General Corporation is proceeding with the design and development of the SNAP-8 Power Conversion System, as authorized by National Aeronautics and Space Administration (NASA) Contract No. NAS 5-417.

The ultimate objective of the SNAP-8 Program is to design and develop a 35-kw electrical generating system for use in various space missions. The power source will be a nuclear reactor furnished by the Atomic Energy Commission (AEC). The SNAP-8 system will use a eutectic mixture of sodium and potassium (NaK) as the reactor coolant and will operate on a Rankine cycle, with mercury as the working fluid for the turbogenerator. It is to be launchable from a ground base and be capable of unattended full-power operation for a minimum of 10,000 hours. After the system is placed into orbit, it is to be capable of activation and shutdown by ground command.

This is the second of two volumes comprising the semiannual materials report submitted in partial fulfillment of the contract. Part of the information appearing in this volume was prepared at Aerojet-General Nucleonics, San Ramon, California, under Aerojet-General Corporation Subcontract 274949. Volume I covers electrical insulation development.

The Component Materials Development Program was under the direction of R. S. Carey, Materials Department Manager, SNAP-8 Division, Von Karman Center. The following engineers contributed to work reported in this volume:

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## GLOSSARY

Abbreviations commonly used in the SNAP-8 Program are defined below.

AA	Alternator assembly	HRL	Heat rejection loop
AEC	Atomic Energy Commission	HRS	Heat rejection system
AGC	Aerojet-General Corporation	HTL	Heat-transfer loop
AGN	Aerojet-General Nucleonics	L/C	Lubricant/coolant
AI	Atomics International	L/CL	Lubricant/coolant loop
AOC	Award of contract	LeRC	Lewis Research Center
ATL	Acceptance test loop	LML	Liquid mercury loop
AZFO	NASA-Azusa Field Office	LMS	Liquid mercury stand
BOD	Beneficial occupancy date	LNL	Liquid NaK loop
CGEST	Cold-gas electrical system test	LOL	Liquid organic loop
CL	Corrosion loop (AGN)	LOS	Liquid organic stand
CPC	Ceramic potting compounds	LPL	Low power loop
CTL	Component test loop (AGN)	MIS	Mercury injection system
DDAS	Digital data acquisition system	Mix-4P3E	Bis(mix-phenoxyphenyl) ether, a mixture of the six possible isomers of bis(phenoxyphenyl) ether; considered as a lubricant/coolant fluid for the PCS
DWG	Drawing		
EDM	Electrical-discharge machining		
EFF	Efficiency		
EGS	Electrical generating system	ML	Pyre-ML, Du Pont polyimide organic resin; as employed in statorette serial numbers, indicates the use of this substance
EM	Electromagnetic		
EME	Electromagnetic equivalent		
FPS	Flight prototype system	MLA	Mercury loop assembly
FPTF	Flight prototype test facility	MN <sub>2</sub> S	Mercury-nitrogen system
FRA	Flight radiator assembly	MPMA	Mercury pump motor assembly
GE	General Electric Company	NaK	Eutectic mixture of sodium and potassium
GN <sub>2</sub> S	Gaseous nitrogen stand		
GPS	Ground prototype system	NASA	National Aeronautics and Space Administration
GPTF	Ground prototype test facility		
HML	Heavy coating of ML ( <u>q.v.</u> )	NF	Nuclear facility
HR	Heat rejection	NHRA	NaK heat-rejection assembly
HRF	Heat rejection fluid	NPA	NaK pump assembly

## GLOSSARY (cont.)

NPMA	NaK pump motor assembly	SL-2	System Loop Test Facility No. 2
NPS	Nuclear power system	SL-3	System Loop Test Facility No. 3
NPSH	Net positive suction head	SL-4	System Loop Test Facility No. 4
NS	Nuclear system	SMU	Structural mockup
NSL	NaK simulation loop	SNAP	Systems for Nuclear Auxiliary Power
ORNL	Oak Ridge National Laboratory	SR	Saturable reactor
PBRF	Plum Brook Reactor Facility	SS	Stainless steel
PCS-1	Power Conversion System No. 1	TA	Turbine assembly
PCS-2	Power Conversion System No. 2	TAA	Turbine-alternator assembly
PCS-3	Power Conversion System No. 3	TAT	Type-approval test
PCS-4	Power Conversion System No. 4	TCL	Thermal convection loop (AGN)
PF	Power factor	TR	Transformer-reactor (assembly)
PL	Primary loop	TS	Test section
PLR	Parasitic load resistor	TRW	Thompson Ramo Wooldridge
PMA	Pump-motor assembly	TSE	Test support equipment
PNLA	Primary NaK loop assembly	VLB	Vehicle load breaker
PO	Purchase order	VR	Voltage regulator-exciter
PTAT	Preliminary type-approval test	W/O	Without
PVT	Pressure-volume-temperature	WOO	Western Operations Office
R <sub>B</sub>	Rockwell B (hardness)	-X	Standing alone (i.e., not preceded by letters of the alphabet), these designations indicate design stages of SNAP-8 hardware
RPL	Rated power loop	-1	
SC	Speed control	-2	
S8DS	SNAP-8 development system	-3	
S8ER	SNAP-8 experimental reactor		
SL-1	System Loop Test Facility No. 1		

SNAP-8 MATERIALS REPORT FOR JANUARY-JUNE 1964  
VOL. II - DEVELOPMENT OF COMPONENT MATERIALS

by H. Derow and B. E. Farwell

Aerojet-General Corporation

SUMMARY

Work was performed during the first half of the 1964 calendar year with the aims of providing data to guide the selection of materials for SNAP-8 system components, and providing metallurgical assistance in the design, development, fabrication, and testing of that system. This work is summarized below under the applicable task categories.

Staff Support

Post-test analyses were performed on two experimental single-tube mercury boilers, each of which operated for approximately 250 hours. One boiler utilized Type 316 stainless steel (SS) mercury-containment tubing, and the other utilized 9% chromium, 1% molybdenum alloy (9Cr-1Mo) steel, the reference alloy for the SNAP-8 boiler and condenser. The 316 SS boiler tube was attacked by the mercury, but no attack on the 9Cr-1Mo boiler tube was discernible. A typical leached corrosion layer was found on the 316 SS boiler tube. It is postulated that this layer, after exposure to air during the changing of a flow-restriction plug in the mercury-tube inlet, prevented thermal rewetting on subsequent restarts. The 9Cr-1Mo steel boiler tube showed no visual or microscopic evidence of surface leaching, or surface dissolution, by the mercury. There was no measurable change in tube-wall thickness.

Mass-transfer corrosion products were found adhering to the interior surface of the 9Cr-1Mo mercury-containment tube in the area of approximately 80 to 100% mercury-vapor quality. The presence of elements strange to the 9Cr-1Mo steel chemistry (9% Cr, 1% Mo, balance Fe) apparently resulted because the loop in which the boiler was tested contained a Haynes 25 condenser and 316 SS piping, and because a 316 SS boiler had been tested previously. The NaK (eutectic mixture of sodium and potassium) side of the mercury-containment tube was found to contain some crevice corrosion and decarburization. These effects are attributed to exposure to unpurified (high-oxygen-content) NaK in a loop without a NaK purification system.

An evaluation of cold pressure welds between nickel and 6061-T6 aluminum was completed. Specimens were evaluated (by means of tension and torsion tests and metallographic study) in the as-received condition and after thermal cycling between 70 and 500°F. The joint was found to be sound.

A series of SNAP-8 specifications were issued and/or upgraded to maintain control of the fabrication and assembly of components within the necessary quality requirements of liquid-metal systems operating unattended for a 10,000-hour life.

Studies were conducted to facilitate the welding of various SNAP-8 system components. A procedure was devised for local stress relief of welds connecting Conoseal flanges to components fabricated from 9Cr-1Mo steel. These welds must be stress-relieved without heating the Conoseal above approximately 500°F to maintain critical flange dimensions for proper sealing. A procedure was devised whereby the weld is heated to the stress-relieving temperature, using an induction heater, and the Conoseal flange and component are protected by water-cooling coils wrapped around the pipe approximately 1/2 in. on both sides of the weld.

#### System Fluids

Procurement specifications were written for the three SNAP-8 fluids: mercury, NaK, and polyphenyl ether (mix-4P3E).

Post-test samples of mix-4P3E fluid were analyzed. They represented two quantities that had crystallized during component-development tests in two separate instances. The fluid, manufactured by the Dow Chemical Company, was found to be especially high in the m-m isomer of the material (92%). Although it is known that a high m-m isomer content results in crystallization, the reason for the reaction is not understood.

#### Boiler Materials and Fabrication

Short-time elevated-temperature and room-temperature tensile tests were performed on welded and unwelded 9Cr-1Mo steel sheet. The unwelded specimens exhibited greater strength at all temperatures (1250 to 1325°F), although all the specimens had been heat-treated together. At 1300°F the ultimate tensile strength of the welded material was approximately 14% lower than that of the unwelded material, and the yield strength was approximately 20% lower. The reason for the difference is not apparent. Welded and unwelded 9Cr-1Mo steel creep-test specimens were partially fabricated.

Through solicitation of proposals it was determined that 9Cr-1Mo/316 SS bimetal tubing can be procured without preliminary fabrication development. Firm quotations were received from two suppliers for bimetal tubing conforming to SNAP-8 quality standards as defined by Quality Level B of Aerojet-General Standard AGC-STD-1161.

Test capsules were completed that contained a 9Cr-1Mo/316 SS transition joint that was fabricated using 310 SS weld-filler metal. Work was started on the first phase of a simulated SNAP-8 operational-environment test to evaluate the adequacy of the joint. In this phase the specimens are subjected to a simulated SNAP-8 startup procedure, during which they are exposed to a nominal temperature of 1325°F and a nominal tube-wall stress of 1100 psi. Thirteen cycles of a scheduled 25-cycle test series were completed during this report period.



Prototype refractory/316 SS bimetal tubes (furnished by Nuclear Metals, Inc.) were evaluated. Pure columbium (Cb) and columbium - 1 zirconium (Cb-1Zr) alloy were used as tube-liner materials. Only the pure Cb/316 SS combination was found to be satisfactory. Nuclear Metals was instructed to proceed with the fabrication of a production quantity of tubing using pure Cb/316 SS. Thermal exposure of flat, multimetal-layered sheet specimens was started. These specimens contain various candidate interface systems that are being considered as means of preventing potentially detrimental diffusion effects and brittle-phase formation at the Cb/316 SS interface. Specimens exposed for 500 and 1000 hours were removed and set aside for later evaluation.

#### Condenser Fabrication

Two -1 model condenser-tube-bundle assemblies (73 tubes welded to tube sheets at each end) were back-brazed using AMS-4778 brazing alloy. Both were adequately precleaned of surface oxide by hydrogen treatment at 1950°F. In both instances, however, some distortion of the condenser-tube bundle occurred. A double brazing cycle was required because the first cycle resulted in unsatisfactory braze joints. After brazing, the joints were checked for helium leakage and were found to be sound.

Work was started on the development of a rolled and welded tube-to-tube-sheet joint to replace the welded and back-brazed joint. The tube-to-tube-sheet weld parameters required modification because the rolling operation, which is performed prior to welding, produced a more intimate fit between the tube and the tube sheet. A slight increase in the weld current produced satisfactory weld penetration. More consistent weld contours and penetrations were produced in welding the rolled tube as compared with the non-rolled design.

#### Turbine Rotor and Nozzle Materials

Eleven different turbine blade and nozzle materials were evaluated for resistance to erosion by elevated-temperature wet mercury. Twenty-one samples of these materials were exposed to 85%-quality vapor at 640°F flowing at 1000 ft/sec. Stellite 6B exhibited the greatest resistance to erosion and was recommended as the material to be used for -3 model turbine rotors and nozzles.

An investigation was conducted to establish an acceptable quality level for ultrasonic inspection of Stellite 6B raw-material blanks for machining turbine rotors and nozzles. A standard was fabricated that incorporated artificial defects of various sizes down to a diameter of 1/32 in. The Haynes Stellite Company detected a 3/64-in.-dia defect but not the 1/32-in.-dia defect. The latter was detected, however, by Automation Industries. Four production articles were inspected by Automation Industries and were found to be free of all defects when compared with the standard. It was concluded that Haynes Stellite was capable of producing material meeting the strictest ultrasonic requirements of Specification AGC-46171 (Class I), even though its inspection procedures and/or equipment did not permit evaluation of the material to this level.

### Mercury-Corrosion-Loop Program

Two mercury pumps satisfactorily completed performance tests in an all-liquid-mercury pump-test loop. The impeller and impeller case of one pump were fabricated from 9Cr-1Mo steel; on the second pump the same parts were made from 405 SS.

Tests of electronic pressure transducers were completed. In addition to obtaining dynamic loop-operation data, it was established that the optimum position of the transducer cooling leg is in the vertical plane. No significant difference in temperature or pressure measurements was noted between transducers with horizontal and vertical cooling legs, but vertical attachment eliminated the possibility of a refluxing action by the mercury vapor.

Mercury Corrosion Loop 3 was assembled and installed in the test cells. The loop was checked out and operated for approximately 407 hours. The NaK in the primary loop was purified to an oxide content of 19 ppm (initial level 68 ppm) through the use of only a cold trap. Hot trapping was found to be unnecessary.

The mercury boiler did not produce superheated mercury vapor immediately on startup. After the loop operated at less than design conditions for approximately 195 hours, superheated mercury vapor was produced, although not at rated SNAP-8 conditions.

The assembly of Mercury Corrosion Loop 4 was initiated.

An impingement corrosion-product separator for use in the superheated-mercury-vapor portion of the SNAP-8 system was designed and fabricated. The conceptual design of a liquid-mercury corrosion-product separator was completed. A cyclone corrosion-product separator was tested in the superheated-mercury-vapor portion of the component test loop and was found to be ineffective in reducing corrosion-product carryover from the boiler. Subsequent examination of the interior of the separator indicated that the experimental conditions did not provide a satisfactory test for the design.

The boiler of the component test loop ceased to produce superheated mercury vapor. A leak developed on the NaK side of the boiler during an attempted "run-in" period, and the loop was shut down.

## I. INTRODUCTION

The objectives of the SNAP-8 Materials Program are to provide data to serve as a basis for the selection of materials for use in various components; to assist, through metallurgical studies, in the design, development, fabrication, and testing of the SNAP-8 system; and to evaluate the resistance of the reference material (i.e., 9Cr-1Mo alloy steel) to mercury corrosion. This report covers the work performed during the first 6 months of the 1964 calendar year.

## II. STAFF SUPPORT

### A. COMPONENT DESIGN AND DEVELOPMENT (TASK A.1)

#### 1. Post-test Analysis of Heat-Transfer-Test Boilers

The TS-1E-1 boiler (tube-in-tube design) was fabricated of 316 SS and was tested to determine the optimum dimensions for a mercury-tube-inlet flow-restriction plug. The test period was 238 hours. The boiler initially performed within design expectations, but the production of superheated mercury vapor ceased as the testing continued. The boiler output was within the design limits (1250°F Hg vapor at 265 psia) during operation with the first two inlet-plug designs. Degradation appeared to have started following a subsequent plug change. Replacement with the initial plug failed to restore the proper performance.

Post-test analysis of the mercury-containment tube revealed a porous internal-surface layer indicative of general leaching-type mercury corrosion. It is postulated that exposure of this layer to air caused a surface oxide to form within the leached area (i.e., in the subsurface voids as well as the tube surface), and that this oxide prevented thermal rewetting on subsequent restarts. The results of this investigation are covered in Ref. 1.

The mercury-containment tube and the internal twisted ribbon of the TS-2B boiler (tube-in-tube design) were analyzed after 269 hours of mercury service. This boiler was fabricated of 9Cr-1Mo steel (the reference alloy for the SNAP-8 boiler and condenser). Deposits of mass-transfer products were found. The heaviest buildup - a maximum of 0.008 in. at one point - occurred in the middle four turns of the eight-turn boiler-tube coil. It is estimated that the vapor quality in this region is approximately 80 to 100%. The mercury inlet and outlet (liquid mercury at approximately 500°F and superheated mercury vapor at approximately 1250°F, respectively) had only a few small localized areas of buildup approximately 0.0002 in. thick.

Emission spectroscopy indicated that the deposit contained Cr, Ni, Fe, and Mn. The presence of nickel was attributed to the operating history and design of the loop system used. A 316 SS boiler was previously tested in this same loop, and the system contained 316 SS piping and a Haynes 25 condenser. Crevice-like attack and decarburization by the NaK on the 9Cr-1Mo tubing were noted.

This attack was attributed to the high oxide content of the NaK, which resulted from operation of the loop with no NaK cold trap for oxygen removal. The results of this investigation are presented in Ref. 2.

Evaluation of this 9Cr-1Mo heat-transfer-test boiler can be utilized to predict some general qualitative effects that can be expected during the operation of a full-scale boiler in a SNAP-8 system. However, the loop system used in testing the TS-2B boiler did not contain all the SNAP-8 components that might have an influence on the results (a turbine-alternator assembly, for example). The loop included materials of construction that will not be included in the SNAP-8 system (e.g., the Haynes 25 condenser and 316 SS mercury-loop piping). Finally, the NaK in the SNAP-8 system will be of low oxygen content (approximately 20 ppm).

A buildup of mass-transfer deposit can be expected on the mercury-containment-boiler-tube wall and on the surface of the twisted ribbon. The buildup found on the TS-2B boiler is shown in Figure 1. The composition, nature, and effects of this buildup on SNAP-8 boiler operation cannot be appraised from the data produced by this test because of the system differences referred to above.

The decarburization and associated grain growth of the 9Cr-1Mo tubing that occurred on the NaK side (see Figure 2) represent a potential problem of reduced creep strength of the material. Potential alternative solutions that eliminate 9Cr-1Mo exposure to NaK are briefly discussed in Sections IV,B and IV,D of this report.

## 2. Design Properties of Materials

Design values for physical and mechanical properties of the 9Cr-1Mo alloy steel (a) normalized at 1700°F and tempered at 1350°F, and (b) annealed 316 SS were distributed within the SNAP-8 project organization. They were selected after a review of published data as well as unpublished test data contained in Ref. 3. Figures 3 and 4 show these properties as a function of temperature.

The linear coefficient of expansion of cement made of fused magnesium oxide and calcium aluminate (Norton Company LM 1625), an encapsulant material for electrical components, was determined in air using a Leitz Dilatometer. Samples were held at 800°F for 64 hours prior to testing, to effect complete dehydration of the material. Sample 1 was tested immediately after dehydration, and Sample 2 was tested after being held 6 hours in a desiccator following dehydration. The results tabulated below were obtained.

Linear Coefficient of Thermal Expansion  
Fused Magnesium Oxide/Calcium Aluminate Cement  
in./in.-°F x 10<sup>-6</sup>

Temp Range °F	Sample 1 (Immediately After Dehydration)	Sample 2 (6 Hours After Dehydration)
80-150	5.71	6.15
80-200	5.75	6.25
80-300	5.77	6.25
80-400	6.16	6.28
80-500	6.31	6.50
80-600	6.44	6.63
80-700	6.53	6.61
80-800	6.67	6.74

### 3. Electrical-Terminal Joint

Pressure welds of nickel to 6061-T6 aluminum alloy were evaluated for possible use in mounting ceramic terminal leaders on electrical components. The specimens were produced by the Utica Turbine Parts Division of the Kelsey-Hayes Company, using its Koldweld process. Tension and torsion tests and metallographic evaluations were performed on as-received and thermally cycled specimens (10 cycles between 70 and 500°F).

The joint was found to be of a metallurgical nature, with a narrow diffusion zone. All mechanical tests produced failure in the aluminum alloy. The results of the investigation are presented in Ref. 4.

### B. COMPONENT FABRICATION (TASK A.2)

#### 1. Specifications

The following specifications were issued or modified during this report period:

Number	Title
AGC-10319	SNAP-8 Components, Cleaning of
AGC-10319/1	Cleaning of Precision SNAP-8 Components and Systems, Procedure for
AGC-10331	Electrical Connections, Inert-Arc Welding of, Procedure for
AGC-10338	Stress Relieving, Local, Welded Tube-to-Flange Joints

<u>Number</u>	<u>Title</u>
AGC-13860 (Class 15)	Quality Level, Radiographic, Fusion Weldments
AGC-14067B	Welders' Qualification Tests for SNAP-8 Program
AGC-10197A	Alloy Steel, Sheet, Strip, Bar, Plate and Forging (9 Chromium - 1 Molybdenum)
AGC-10227A	SNAP-8 Materials, Inert-Arc Welding of, Procedure for
AGC-10335	FEP-Fluorocarbon Resin Coating
AGC-10226B	Steel, Cleaning and Dehydration, Procedures for

## 2. Turbine-Alternator Assembly (TAA)

The Palmer Company, Whittier, California, encountered difficulties in the fusion butt welding of 0.020-in.-thick 9Cr-1Mo sheet for use in the turbine-exhaust bellows. Visible weld defects occurred in the form of small nodules on both the face and the root side of the weld. Radiographic and metallographic examination showed the welds to be of excellent quality despite the visible defects. The surface condition of the base material was judged to be unsatisfactory, and mechanical polishing or electropolishing was recommended to correct the condition. Subsequent welded samples prepared with abrasive-belt-sanded material exhibited a much improved weld-surface contour, but the problem has not been resolved satisfactorily. Additional work is under way.

## 3. Turbine Simulator

Weld samples were prepared at Aerojet's Von Karman Center to determine the weld penetration of turbine-simulator (9Cr-1Mo steel) tube-to-tube-sheet joints as a function of welder-current setting. The joint configuration is shown in Figure 5. Sections welded at 40 amp showed 0.025- to 0.032-in. penetration along the faying surfaces. Sections welded at 70 amp showed 0.085- to 0.093-in. weld-joint penetration. The weld penetration of the second sample exceeded 1-1/2 times the tube-wall thickness (0.050 in.), and 70 amp was recommended as a proper current setting.

## 4. -1 Model Boiler

The boiler fabricator is Western Way Manufacturing Company (WW), Van Nuys, California. The mercury side of the boiler is made of 9Cr-1Mo steel. The major fabrication difficulty was completion of the tube-to-tube-sheet joints using a TIG automatic internal-tube-welding procedure.

Preliminary tube-to-tube-sheet joint development had been performed by WW using Type 1010 steel tubes and 9Cr-1Mo tube sheet, because 9Cr-1Mo

tubing of the proper diameter was unavailable. The weld parameters thus developed were not suitable for use on an all 9Cr-1Mo joint. As a result, the automatic internal tube welds on the Serial No. A-1 and A-2 boilers were erratic in both penetration and contour and contained extensive linear porosity.

The weld-technique development work was subsequently successfully concluded with only 9Cr-1Mo steel. The tube-to-header joint design that was evolved is shown in Figure 6. The abutting ends of the tube and tube sheet were polished with fine emery paper prior to welding. Parts were then scrubbed with a stainless-steel wire brush and washed with trichloroethylene (TCE). It was also necessary to make several minor changes in the welding equipment, a proprietary design of WW. The newly developed procedures are ready for use on the Serial No. A-3 and A-4 boilers, which are also being fabricated by WW.

The internal surfaces of the mercury-containment tubes were degreased at WW in a forced TCE flush prior to a final stress-relief operation. The procedure developed is described in Ref. 5 (pp. VIII-2 and -3).

#### 5. NaK Pump-Motor Assembly (PMA)

Weld-cracking difficulties were encountered at the Mechanical Specialties Company, Los Angeles, in the fabrication of the rpm-signal-generating section of the rotor for the NaK PMA. The rotor consists of an Inconel disk with slots that are weld-filled with mild-steel wire. A procedure was developed at the Von Karman Center that resulted in satisfactory welds. Because of the high cracking sensitivity of the material combination, however, it was recommended that a non-welded design be utilized.

#### 6. Localized Stress Relief of Conoseal Flange Welds

Stress relief is required at 9Cr-1Mo steel weld joints that attach Conoseal flanges to component piping. The Conoseal flange (and in some cases the component) must not be heated to the stress-relieving temperature (1350°F), due to the danger of heat distortion. Methods of localized stress relief were therefore investigated.

It was experimentally determined that the joints could be stress-relieved by means of induction heating and local cooling. A water-cooling coil will keep the temperature of the tube under 500°F approximately 0.5 in. from the weld, thus preventing heat transmission to the Conoseal or the component.

To ensure adequate temperature control and prevent overheating of the weld area, thermocouples are employed during the stress-relief treatment. Several types were investigated, using a spot-welded thermocouple as a reference standard up to 1200°F and an optical pyrometer as the standard above 1200°F. Inconel spring-loaded thermocouples were found to indicate temperatures to 900°F accurately. Wire-wrapped contact couples consistently indicated a temperature 80°F lower at 1350°F. Capacitance-discharge-welded couples and an optical pyrometer indicated temperatures within 10°F of each other, up to 1350°F.

Specification AGC-10338 was written to cover localized stress relieving of 9Cr-1Mo weld joints using capacitance-discharge-welded thermocouples as temperature indicators. The 9Cr-1Mo, Conoseal flange joint on the A-2 boiler was locally stress-relieved successfully by this procedure.

### III. SYSTEM FLUIDS (TASK A.4)

#### A. PROCUREMENT SPECIFICATIONS

The specifications issued to cover the procurement of mix-4P3E, mercury, and eutectic NaK for non-nuclear test operations are as follows:

<u>Number</u>	<u>Title</u>
AGC-10320	Polyphenyl Ether, Mix-4P3E
AGC-10327	Mercury, Heat-Transfer Grade
AGC-10340	Sodium-Potassium Eutectic (Commercial Grade Purity)

Commercial-grade NaK is being bought in lieu of an available high-purity grade. It is produced with no special control over carbon, oxygen, calcium, and other impurities, and is certified by the vendor to consist of 22  $\pm$ 1% Na and 78  $\pm$ 1% K. High-purity NaK, on the other hand, is analyzed using a sample taken at 600°F and is guaranteed to contain trace impurities amounting to 1000 ppm (maximum total) and carbon, oxygen, and calcium to a maximum of 50 ppm each. It would have essentially commercial-grade purity with respect to oxygen content after loading into the loop, due to reaction with the oxide surface layer normally present on all metals exposed to air. The NaK would thus have to be purified to remove excess oxygen, regardless of its purity when loaded into a loop system. The calcium content is not considered significant in a non-nuclear test environment. The carbon content is not considered to significantly affect corrosion mass transfer or carbon transport in the SNAP-8 system.

#### B. MIX-4P3E, SNAP-8 LUBRICANT/COOLANT FLUID

All work performed in the SNAP-8 program in evaluating the mix-4P3E polyphenyl ether lubricant/coolant fluid is summarized in Ref. 6. Included are evaluations of foaming characteristics, purity determinations and purification-technique development efforts, and electrical properties after extended elevated-temperature exposure, as well as cursory compatibility tests with mercury, NaK, and metallic and nonmetallic materials. (Dow ET-378 was used in this study.)

In the current report period, mix-4P3E polyphenyl ether crystallized during component-development tests. Crystallization was encountered at the General Electric Company, Pittsburgh, Pennsylvania, during alternator tests and at Aerojet's Von Karman Center during pre-test operation of a gaseous-nitrogen loop to be used for TAA testing. An analysis was performed on fluid samples by means of gas chromatography. The crystallized residue was found to have an especially high content (92%) of the m-m isomer of mix-4P3E. Analytical curves supplied by the Dow Chemical Company of Midland, Michigan on the basis of gas chromatography of a production run of Dow's mix-4P3E has the following analysis:



<u>Isomers of Mix-4P3E</u>	<u>Wt%</u>
m-m	73.2
p-m	18.7
o-m	7.5
p-p	0.5
o-o	0.1
o-p	None identified

This composition, as well as the m-m isomer content of the fluid sample, differs considerably from the calculated eutectic composition (Ref. 7), which is as follows:

<u>Isomers of Mix-4P3E</u>	<u>Wt%</u>
m-m	45
p-m	35
o-m	10
o-p	5
p-p	1 or less
o-o	1 or less

Deviation from the calculated eutectic range is known to result in eventual crystallization. It is not known what initiates the crystallization in the supersaturated condition.

#### IV. BOILER MATERIALS AND FABRICATION

##### A. 9Cr-1Mo WELD (TASK D.1.a)

The objective of this task is to establish the gross strength efficiency of 9Cr-1Mo steel welds by elevated-temperature, tensile, and creep-rupture tests at 1325°F, the approximate maximum operating temperature of the boiler.

Welded and unwelded base-metal tensile-test specimens (see Figure 7) were prepared from 1/8-in.-thick 9Cr-1Mo steel sheet. The material was purchased from Republic Steel Corporation and conformed to Specification AGC-10197. The welds were not machined but contained a crown, on both sides, of approximately 1/64 in. The weld thus represented the typical condition of a butt-welded mercury-containment tube for boiler use.

The unwelded base-metal specimens were heat-treated in argon by normalizing at 1700 +25°F for 45 min, with air cooling followed by stress relief in argon at 1350 +25°F for 1 hour and air cooling to room temperature. The welded specimens were normalized with the base-metal specimens prior to welding. They were then welded in accordance with Specification AGC-10227,

using a preheat temperature of 400°F. The welded assemblies were allowed to cool slowly to room temperature in the welding fixture. They were then placed in a furnace at 400°F over a weekend because it was not possible to stress-relieve within 12 hours after welding, as required by the welding specification. The welded specimens were subsequently stress-relieved with the unwelded base-metal specimens. X-ray and dye-penetrant methods were used to inspect the weld, which conformed to the Class 15 radiographic quality level of Specification AGC-13860. Tensile tests at elevated temperature in air were performed and the results are given in Table 1.

Failure of all specimens, welded and unwelded alike, occurred in the parent metal. On visual inspection, the welds did not appear affected by the applied load (i.e., no elongation across the weld was apparent). However, elongation and reduction of the cross-sectional area of the welded specimens occurred at both edges of the weld zone. The location of the failure was far from the weld and the weld-heat-affected zone.

The unwelded specimens consistently exhibited higher ultimate tensile and yield strengths than the welded specimens, even though the failure was in no way associated with the weld area. As an illustration, at 1300°F the tensile strength of unwelded 9Cr-1Mo steel was 14% higher than the welded and the 0.2% yield strength was 20% higher. There is no apparent reason for this difference.

Welded and unwelded (base-metal) creep-test specimens, using the same 9Cr-1Mo sheet as above, were partially completed as of the end of this report period. The specimens are being tested in air at 1325°F. The stresses imposed on the specimens are 1600 and 2300 psi. The creep test is scheduled to last 3000 hours.

#### B. 9Cr-1Mo/316 SS BIMETAL TUBE (TASK D.1.d)

The objective of this task is to establish the availability of a bimetal tube comprised of nonrefractory, conventional materials. This tubing will be evaluated to establish its utility as a backup material to the 9Cr-1Mo steel mercury-containment tube for boiler service.

Based on evaluation of the TS-2B heat-transfer-test boiler (see Section II,A,1) and preliminary test data developed by the Oak Ridge National Laboratory, it appears that the 9Cr-1Mo steel mercury-containment tube in the SNAP-8 boiler will be decarburized on the NaK side, and marked grain growth will occur in the decarburized zone. This phenomenon is caused by carbon transport from the 9Cr-1Mo steel, through the NaK, to the 316 SS in the system (316 SS is used for the boiler shell and the piping of the NaK primary loop). This may result in an unacceptable reduction in mechanical properties. The 9Cr-1Mo tubing can be protected against such attack through the use of a bimetal mercury-containment tube. The tube would consist of a 9Cr-1Mo steel liner clad with 316 SS to eliminate contact between the NaK and the liner material.

An Advanced Quotation Request was initiated to determine the availability, cost, delivery schedule, and quality level of 9Cr-1Mo/316 SS bimetallic tubing for boiler applications. Two vendors proposed to supply such tubing conforming to Quality Level B of Aerojet Standard AGC-STD-1161. In 300-ft quantities, the prices per foot were \$40 and \$65.

The structure of this bimetal tube is based on an assumption that 9Cr-1Mo steel provides mercury-corrosion resistance only and that 316 SS provides the required elevated-temperature strength. Because several alternate alloy combinations will fulfill these requirements, an evaluation of other combinations was initiated.

#### C. 9Cr-1Mo/316 SS TRANSITION JOINT (TASK D.1.e)

The objective of this task is to evaluate welded transition joints connecting 9Cr-1Mo steel and 316 SS using 310 SS weld-filler metal. Such a joint is required at various interfaces between the first three loops of the SNAP-8 system. The joint must withstand extended service at temperatures to approximately 1325°F (the mercury-superheated-vapor manifold of the boiler).

Transition joints will be exposed to a simulated SNAP-8 environment in a two-phase test program (see Figure 8) to evaluate the adequacy of the joint. The stress will be produced by internal pressurization of a test capsule. Four test capsules containing a transition joint were fabricated (see Figure 9). The welds were made in accordance with Specification AGC-10227; all passed dye-penetrant inspection and conformed to the Class 15 radiographic standard of Specification AGC-13860. The capsules were stress-relieved after welding at 1350 ± 25°F in argon for 1 hour and were air-cooled.

The welds of two capsules were roll-formed by the Super-Temp Corporation, Santa Fe Springs, California. The weld thickness was reduced 25% by rolling at 1700°F to break up the weld microstructure. These capsules were subsequently heat-treated again by normalizing in argon at 1700 ± 10°F for 1 hour and tempering at 1500 ± 25°F for 30 min. The latter temperature was selected to achieve full-grain refinement of the hot-worked weld structure. The two capsules were X-rayed. One showed shear cracks in the heat-affected zone of the weld and was rejected; the other conformed to Class 15 of Specification AGC-13860.

The outer surfaces of the three acceptable capsules were chromium-plated, and gage marks were applied for use in the measurement of creep across the weld joint during test exposure.

The first phase of the evaluation program (SNAP-8 startup-cycle simulation) was partially completed. Thirteen of 25 programmed 8-hour cycles were completed. The program was interrupted by the failure of furnace heating elements. Replacement elements were procured and were being installed at the end of the report period.

D. REFRACTORY-BIMETAL TUBING (TASK D.1.f)

The objective of this task is to establish the availability of refractory-bimetal tubing as a backup material for mercury containment in the SNAP-8 system if experience indicates that 9Cr-1Mo steel does not exhibit sufficient mercury-corrosion resistance for 10,000-hour life. If direct-bonded tubing (refractory to 316 SS) cannot be fabricated or proves to be unusable because of elevated-temperature diffusion effects, a source is to be developed for refractory-bimetal tubing fabricated with an interface material or materials between the refractory and the 316 SS.

The evaluation of three prototype refractory-bimetal tubes furnished by Nuclear Metals was completed. No bond defects were found in a pure Cb/316 SS tube before or after coiling to an 8-in. diameter. Two tubes of Cb-1Zr/316 SS were found to contain gross unbonded areas in the as-received condition and to be completely unbonded in the outer fiber after coiling. Metallographic and ultrasonic inspection methods were employed. Nuclear Metals was authorized to complete an outstanding order for bimetal tubing to consist of pure Cb and 316 SS. The applicable technical requirements are appended.

Thermal exposure of pure Cb/316 SS tubing specimens from Nuclear Metals and flat bonded sheet specimens (described in Ref. 8, p. VIII-27) from Metals & Controls, Inc., Attleboro, Massachusetts, was started at the Materials Testing Laboratories, Los Angeles. These specimens will be evaluated for diffusion effects resulting from elevated-temperature exposure. The test environments are  $1350 \pm 10$  and  $1450 \pm 10^\circ\text{F}$  under a vacuum of approximately  $4 \times 10^{-6}$  mm Hg. Sets of specimens exposed for 500 and 1000 hours were removed and set aside for later evaluation. Exposure of the remaining specimens continued toward the goal of 2500 hours.

E. REFRACTORY-BIMETAL BRAZING (TASK D.2.b)

The objective of this task is to develop techniques of joining refractory-bimetal tube to refractory-bimetal tube sheet for boiler applications by means of back-brazing procedures. The bimetal-alloy system consists of pure Cb and 316 SS, and the Pyromet Company, San Carlos, California, initiated a program to attain these objectives.

During this report period, Pyromet started fabrication of the refractory-bimetal, pure Cb/316 SS, tube sheet. The Horton-Clad bonding process will be used. By means of this proprietary process of Chicago Bridge and Iron Company, two or more metals are bonded by exposing the assembly composite, with a braze alloy between the surfaces to be bonded, to a high temperature under vacuum. Pyromet also started work on designing the tube-to-tube-sheet joint. Aerojet shipped a quantity of Cb/316 SS bimetal tubing to Pyromet for use in this program.

V. CONDENSER TUBE-TO-TUBE-SHEET JOINT (TASK D.2.a)

The objectives of this task are to select an optimum joint design and develop an adequate fabrication procedure for the tube-to-tube-sheet joint of the SNAP-8 condenser. The condenser-tube and tube-sheet assembly (see Figure 10) consists of 73 parallel tubes approximately 60 in. long. The ID of the tubes tapers from 0.5 in. at one end to 0.250 in. at the other. To permit parallel alignment of the tubes, the tube sheet at the large-tube-ID end is somewhat larger in diameter than the sheet at the opposite end. Two candidate joint configurations were evaluated during this report period: (a) welding and back brazing, and (b) internal tube rolling followed by welding.

A. WELDED AND BACK-BRAZED JOINT

This joint is completed by first welding the 73 tubes to the tube sheets at both ends (Ref. 9 discusses the welding procedure), followed by back brazing to fill the space between the faying surfaces of the tube and the tube sheet that lies under the weld. The braze reduces the notch effect at the root of the weld. This notch cannot be avoided, because the weld does not penetrate through the tube-sheet thickness due to the large ratio between the tube-sheet and tube-wall thicknesses. The braze coincidentally provides additional strength to the joint.

An evaluation of brazing alloys was completed during this report period. The AMS-4778 braze alloy (Microbraz 130, consisting of 3.5% B, 4.5% Si, 0.20% C maximum, balance Ni) was selected. Joints brazed with this alloy, using minimum gaps between faying surfaces, exhibited shear strengths comparable to those attained with the other alloys. The brazing temperature, however, is significantly lower - a decided advantage in brazing a structure as prone to distortion as the condenser-tube bundle. Comparative test data and associated properties for the materials evaluated are given in Table 2. The evaluation program is covered in Ref. 10 (pp. VIII-24 to VIII-27) and Ref. 11 (pp. VIII-20 and -21).

Several subscale configurations of the condenser-tube and tube-sheet assembly and an experimental full-sized assembly were welded and back-brazed using the AMS-4778 alloy for the purpose of brazing-technique development. This work is covered in Ref. 11 (pp. VIII-21 through VIII-24).

Two -1 model condensers were back-brazed. Excessive distortion of the tube bundle occurred during the brazing of the experimental condenser (see Ref. 11, pp. VIII-24 and -25); a brazing fixture was therefore designed and fabricated. The condenser hangs free from the small header under a dead load of 100 psi on each tube (Figure 10). A heat-radiation shield surrounded the tube bundle during hydrogen cleaning and brazing to minimize the temperature differential within the tube bundle (Figure 11).

The first of these two condensers was processed through a hydrogen cleanup (reduction of surface oxide) and brazing cycle. Table 3 describes the

full process and the incremental time required for the individual steps. The hydrogen treatment did not fully clean the part and resulted in slight distortion of the tube bundle. This was attributed to inadequate hydrogen-transfer rates within the radiation shield. The maximum temperature differential between the outer periphery and the center of the tube bundle was 35° F at 1300° F.

After the first cleanup cycle was completed and before brazing, the hydrogen distribution within the retort was altered to provide a direct flow of part of the hydrogen inside the radiation shield. After brazing, the first condenser exhibited areas of inadequate joint filling, filleting, and wetting, and evidence of incomplete melting was found at the outer edge of the braze alloy on the large header (lower) end. These difficulties were attributed to an insufficiently low hydrogen dewpoint at the joint areas, caused by inadequate movement of hydrogen inside the radiation shield. The rate of hydrogen transfer inside the radiation shield was increased by introducing more hydrogen into this area and venting the shield by drilling 1/4-in.-dia holes, 3 in. on centers, every 60° around the shield for its full length. Additional braze-filler alloy was applied to the areas that exhibited poor wetting, and the part was rebrazed. After the second brazing cycle, the joints were adequately filleted, but excess braze alloy was present and resulted in runoff down several of the tubes.

The second condenser was back-brazed by the ultimate procedure established for the first -1 model condenser (see Table 3). Slight bowing of the tubes occurred once again. A few unfilleted gaps existed at the large header at the bottom, and the gap filling and filleting were inadequate at the small header at the top. The assembly was reversed (putting the large header at the top), a small amount of braze alloy was added, and the assembly was rebrazed at 1950° F. Visually satisfactory brazed joints resulted. Helium-leak checking of the units indicated no leakage through the welded and back-brazed joints.

#### B. ROLLED AND WELDED JOINT (TASK D.2.a)

This joint design is under study as an alternate for the welded and back-brazed joint used on the first two -1 model condensers. Elimination of the back-brazing process is desirable because of difficulties incurred in obtaining consistent braze results and because of very high fabrication costs. The development of the actual rolling procedure is discussed in Ref. 5 (pp. VI-9 and -10).

It was expected that the use of a rolled tube might result in a change in the welding procedure previously established for joining an unrolled tube to the tube sheet, and an appropriate study was performed. The only change found necessary was an increase in the welding current. Satisfactory welds were then produced consistently. Welding a previously rolled tube having an inherently good joint fitup with the tube sheet results in weld joints that are more uniform in contour and penetration than those made in a non-rolled assembly.

VI. TURBINE ROTOR AND NOZZLE MATERIALS (TASK E.1)

The objective of this task is to evaluate candidate turbine blade and nozzle materials for their resistance to erosion by wet mercury vapor at elevated temperatures. Material samples were exposed to accelerated erosion testing in the SNAP-8 low power loop to provide a basis for the recommendation of a material for use in the SNAP-8 turbine assembly.

A total of 21 samples, of 11 different materials, were exposed to the following approximate mercury-vapor conditions: 85%-quality vapor at 640°F flowing at 1000 ft/sec. Tables 4, 5, and 6 and Figure 12 provide descriptive summaries for the materials evaluated and the test results. This program is discussed in detail in Refs. 12 and 13. The results indicated that Stellite 6B, a cobalt-base alloy, provided the greatest resistance to wet-mercury-vapor erosion; it was therefore recommended as the material to be used for -3 model turbine rotors and nozzles.

When efforts were initiated to procure Stellite 6B material for the fabrication of -1 model turbine-rotor and nozzle parts, it was found that suppliers were unwilling to quote on plates and forgings of this alloy to the Class I ultrasonic-inspection quality level of Specification AGC-46171 (Process Specification, Ultrasonic Inspection Acceptance Levels); this quality level allows a maximum defect size of 1/32 in. The unwillingness encountered was based on the unavailability of background information and of previous test experience applicable to this specific alloy. It was accordingly deemed necessary to conduct a study to establish criteria for ultrasonic inspection and to determine a realistic quality level.

Haynes Stellite produced (a) a standard Stellite 6B block with drilled holes of varying sizes down to a 1/32-in. diameter, and (b) four Stellite 6B plates (18 by 3-1/2 by 3/4 in.), typical of the product purchased for the fabrication of SNAP-8 turbine wheels and nozzles. The plates and standard block were ultrasonically inspected by the Haynes Stellite Quality Control Laboratory. The 1/32-in.-dia hole in the standard block was not detected, but a 3/64-in.-dia hole was. The four plates revealed no indications of defects, based on the standard, and they were certified to meet the Class II quality level "or better" of Specification AGC-46171. This class allows a maximum defect diameter of 3/64 in. as determined by ultrasonic inspection.

The plates and the standard block were subsequently reinspected ultrasonically by Automation Industries, Los Angeles. This laboratory detected the 1/32-in.-dia hole in the standard. No defects were detected in the four plates.

It was concluded that the Haynes Stellite ultrasonic-inspection equipment and/or procedure did not permit inspection of the Stellite 6B material to Aerojet's strictest requirements but that the firm is able to produce material meeting those requirements. It was recommended to the turbine-assembly development group that consideration be given to upgrading the quality requirements for Stellite 6B alloy to be used on turbine hardware from the presently specified Class II to Class I of Specification AGC-46171.

## VII. MERCURY-CORROSION-LOOP PROGRAM (TASK C.1)

The objectives of the corrosion-loop program are to determine corrosion and mass-transfer patterns in the mercury and NaK loops of the SNAP-8 system, to evaluate the corrosion resistance of the SNAP-8 reference materials against the 10,000-hour-life requirement, and to develop and test mercury-corrosion-product separators that will remove solid particles from the liquid mercury and superheated mercury vapor. The program is being conducted at Aerojet-General Nucleonics (AGN), San Ramon, California.

Component Test Loop 2 (CTL-2), constructed of Haynes 25 alloy, is being operated to check the performance of certain components to be used in Corrosion Loops 3, 4, and 5 (CL-3, 4, and 5), which are being constructed to 1/16th scale of the SNAP-8 system. The mercury loop will be fabricated of 9Cr-1Mo steel. The NaK primary loop will be constructed of 316 SS, with chromized Hastelloy N in the high-temperature area and a section of Hastelloy C in the low-temperature area. The use of the Hastelloy alloys is dictated by the materials used in the reactor of the SNAP-8 system.

### A. TESTING OF LOOP COMPONENTS

Mercury pumps and pressure transducers to be used in CL-3, 4, and 5 were tested to determine their operating characteristics. An all-liquid pump-test loop was operated for checkout and endurance runs of the mercury pumps to be used in CL-3, 4, and 5. CTL-2 was used for pressure-transducer tests.

#### 1. Mercury Pumps

A mercury pump, Chempump Model CF 7-1/2, with Type 405 SS used for the impeller and impeller case, was installed in a mercury-pump-test loop and operated continuously for 1073 hours under the following typical conditions:

<u>Location</u>	<u>Pressure, psig</u>	<u>Temp, °F</u>
Pump outlet	670	595
Pump inlet	35	435

A second mercury pump, Chempump Model CF 7-1/2, with 9Cr-1Mo steel used for the impeller and impeller case, was installed in the pump-test loop and was operated for 8 hours to check its performance. Both pumps operated satisfactorily and were removed and installed in parallel in CL-3.

#### 2. Pressure Transducers

Seven electronic pressure transducers with two types of cooling legs, horizontal and vertical, were installed in CTL-2, as shown in Figure 13, to obtain dynamic data on loop operation and to evaluate the transducer cooling legs. Two test runs were completed.



The pressure instrumentation for CTL-2 Run 10 consisted of five Statham pressure transducers (0 to 500 psia) and two Consolidated Electrodynamics Corporation (CEC) pressure transducers (0 to 100 psia and 0 to 700 psia) that were excited by two bridge-balance units. The outputs of the pressure transducers were fed through amplifiers and recorded on a Visicorder oscillograph. Valid pressure data could not be obtained during the run because of interactions between channels in the system. These interactions resulted from the use of two bridge-balance units. The 100-psia CEC transducer was removed, and the bridge-balance units were replaced with a Microdot unit for each transducer (this system will be used on CL-3). Another run was then made. Transducer pressure and temperature data from Run 11 are presented in Table 7; other observations are summarized below.

a. The frequency on the CEC transducer at the mercury-pump outlet was approximately 480 cps, with a maximum peak-to-peak amplitude of 45 psi.

b. A frequency of approximately 160 cps and a 10-psi peak-to-peak amplitude were noted on the Statham transducer at the boiler inlet. All other Statham transducers registered this frequency periodically, but at a decreased amplitude.

c. Variance in the boiler-outlet pressure with a peak-to-peak amplitude of about 20 psi was noted on the three Statham transducers located between the boiler outlet and the adjustable choke nozzle. The period of the fluctuation varied between 5 and 25 sec.

d. The rated pressure was established in approximately 1/2 sec after the mercury pump was turned on.

e. The pressure dropped to zero in about 1 sec when the pump was shut off.

f. The adjustable choke nozzle smoothed out the boiler fluctuations and maintained a steady output pressure.

Temperature profiles of the cooling legs and the transducers are shown in Table 7. No significant difference in temperature or pressure between the horizontal and vertical cooling legs was noted; however, the vertical attachment eliminated the possibility of mercury-vapor condensation and return to the loop as liquid. Consequently, vertical attachment of transducers will be used for CL-3.

## B. CORROSION LOOPS 3, 4, AND 5

### 1. Design

Ref. 14 summarizes the mechanical and thermal design of the corrosion loops. A technical memorandum (Ref. 15) was published during this

report period to provide detailed procedures for corrosion-loop checkout, calibration, loading, and startup.

## 2. Fabrication

### a. Components

All components for CL-3, 4, and 5 were completed except for the mercury condenser (Dwg No. 405805),\* the turbine-simulator heat exchanger (Dwg No. 405099),\* and the NaK purification system for CL-5. All components purchased from outside vendors were received.

Difficulties were encountered in the construction of turbine-simulator heat exchangers because of inclusions in the 9Cr-1Mo bar stock from which the end adapters were machined. Helium-leak tests conducted after post-heat treatment of the welds for 30 min at 1350°F revealed porosity. Using 9Cr-1Mo weld wire, a weld bead approximately 0.060 in. thick was applied over the surfaces of the adapters. The weld was heat-treated. The turbine-simulator heat exchanger passed the helium-leak test after the weld bead was placed on the adapters.

### b. Instrumentation

The control console was checked out, thermocouples and pressure transducers were installed on CL-3, and the interconnections between the loop and the control console were completed. The checkout and calibration of all instrumentation, as outlined in Ref. 15, were completed.

### c. Assembly

The assembly of CL-3 in Cells A and B (Figures 14, 15, and 16) was completed, and the loop was installed in the test cells. All welds made during the assembly were X-ray-inspected, and the NaK primary system, the mercury system, and the NaK condensing system were satisfactorily helium-leak-tested. The final hookups of the cover-gas system, cooling water, and electrical power were completed.

## 3. Operation of CL-3

After the initial checkout of CL-3 was completed, NaK circulation was started in the primary system on 2 March 1964. The electromagnetic (EM) pump failed after 1.5 hours of operation because of a crack in the pump-cell tubing that caused NaK leakage. The defect, requiring pump replacement, was apparently due to faulty tubing. Two other repairs were necessary in the NaK primary system of CL-3 before successful system operation was possible:

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\* Drawings in Ref. 14.

a. The NaK expansion tanks required modification to increase the OD of the argon lines (from 1/4 in. to 3/4 in.) that supplied cover gas to the tank. Additionally, the position of a rupture disk in this portion of the loop was changed. Modification was necessary because the smaller cover-gas lines became clogged, causing the NaK to bypass the expansion tank through the line leading to the rupture disk.

b. Two bellows-sealed valves in the NaK purification system became inoperable. The valves were replaced. To avoid further difficulty, the operating mechanisms of all valves were lubricated with a high-temperature lubricant (Silver Goop, Crawford Fitting Company, Cleveland, Ohio).

The NaK in the primary loop of CL-3 was purified by using the cold trap only. The use of the hot trap was not considered necessary, because the oxide level, as measured with the plugging indicator valve, was reduced to 19 ppm by the cold trap alone. The initial oxide level in the NaK was 68 ppm after hot circulation and prior to purification. After the NaK system was cold-trapped for 4 hours with a cold-trap temperature of 250°F, the oxide level was determined to be 30 ppm. The NaK system was cold-trapped again for 6 hours, and the oxide level was found to be 19 ppm.

Mercury boiling in CL-3 was started on 21 April, with the procedure outlined in Ref. 15. The NaK outlet temperature of the boiler was maintained at 1150°F (50°F higher than the design value) to ensure that suppressed mercury boiling did not occur. The loop was shut down after 1.5 hours of operation because of an electrical problem with the saturable reactor that controls the NaK heater. It was necessary to place a step-up transformer ahead of the saturable reactor to increase the voltage to the NaK heater.

Mercury boiling was started again on 25 April, and the loop was operated for 70 hours. The run was terminated when an excessive pressure drop across the condenser was noted. Superheated mercury vapor could not be obtained from the mercury boiler during this run, apparently because the heat flux from the NaK to the mercury was lower than predicted. No explanation for the low heat flux in the mercury boiler can be found at this time.

The excessive pressure drop across the condenser was determined to have been caused by an obstruction in the tapered condenser tubes. The obstruction was removed from the condenser tubes by cutting the inlet and outlet tubing to the condenser and wire-brushing each tube. Upon analysis, the material that caused the obstruction was tentatively identified as shop wiping paper that had inadvertently been left in the condenser inlet when the loop was assembled.

The condenser inlet and outlet tubes were rewelded, heat-treated, and inspected.

The loop was started on 17 May; superheated mercury vapor was not obtained for some time. After a total of 195 hours of operation with no evidence of superheated mercury vapor, a sudden increase in the mercury-boiler

outlet temperature indicated that superheat conditions were achieved. The temperature increase occurred when the loop was operating stably. The adjustable choke nozzle had been opened slowly to lower the boiler outlet pressure over an 8-hour period just prior to the first indication of improved boiler performance. The initial increase in the mercury-boiler outlet temperature (70°F) continued for 1 hour, and then a gradual improvement in boiler performance occurred. The operating conditions before and after the improved boiler performance are summarized in Table 8.

No explanation for the sudden increase in boiler performance can be given at this time; previous experience, however, has indicated the need for a "run-in" period for some forced-convection mercury boilers before superheated mercury vapor is produced.

The maximum boiler outlet temperature recorded was 1205°F with a boiler outlet pressure of 270 psia (rated conditions 1265°F, 265 psia). A mercury flow disturbance occurred during the run and caused a 10-hour shutdown of mercury flow. When mercury boiling was resumed, the boiler did not achieve superheat until 3 hours after boiling was started. The boiler outlet temperature did not reach the 1205°F previously obtained (see Table 8).

The loop was shut down upon failure of the differential-pressure transducer that measures the pressure difference across the venturi flowmeter. The loop operated a total of 407 hours during this report period.

#### C. CORROSION-PRODUCT-SEPARATION STUDY

The purpose of this task is to develop and test mercury-corrosion-product separators that will remove solid particles from liquid mercury and superheated mercury vapor. An impingement type of vapor-phase corrosion-product separator and a liquid-phase separator were designed during this report period, and a centrifugal type of vapor-phase separator was tested in CTL-2.

##### 1. Design

##### a. Vapor-Phase Separators

##### (1) Design Considerations

For the design of a vapor-phase corrosion-product separator, the size and composition of the undesirable particles must be known but in practice they are not known. It has been postulated that mercury droplets that are entrained in vapor and contain dissolved corrosion products cause corrosion-product buildup on the nozzle. Because the vapor is superheated, the mercury droplets will evaporate continuously until equilibrium is reached. Equilibrium may be characterized by liquid mercury containing corrosion products or dry corrosion-product powder. Equilibrium may never be reached, however, because a limited time is required for the droplet to travel from the boiler section to the nozzle. Either dry corrosion products or corrosion products in the mercury

droplets, or both, may therefore accumulate on the nozzle. Even less is known about the size of the particles; for the design of the prototype separators, particle diameters are assumed to be in the micron range. Tests of the separators will then yield some information on particle size.

## (2) Impingement Type

An impingement separator (Figure 17) was designed for testing in CTL-2. The separator (fabricated by Mectron Industries) consists of a pipe with 115 impingement screens at right angles to the mercury-vapor flow; it is intended to remove entrained mercury droplets containing corrosion products. The design of this separator is discussed in Ref. 11 (paragraph VIII,B,1,c).

The areas of greatest uncertainty in the design calculations are the prediction of impact efficiency and the calculation of pressure drop. No way of substantiating the extrapolation appears feasible other than an experimental program; however, pressure measurements along CTL-2 should indicate the accuracy of the pressure-drop calculations.

## (3) Cyclone Type

The design of the cyclone-type vapor-phase corrosion-product separator is covered in Ref. 8 (p. 20).

### b. Liquid-Phase Separator

The liquid-phase corrosion-product-separator design was based on the results of the 9Cr-1Mo thermal-convection-loop tests described in Ref. 16 (Section IV,B,3). The separator will be located immediately downstream of the condenser in CL-4. It consists of two sections (see Figure 18), the first containing 4.06 lb of iron wool and the second 8.32 lb of columbium wool. The size of the separator was determined by available standard pipe sizes and the desire to have an average mercury velocity of  $5 \times 10^{-3}$  ft/sec and a residence time of 300 sec. The pressure drop has been estimated to be less than 1 psi for each section. The specifications for the liquid-phase separator are as follows:

Loop flow rate, lb/hour	600	
Mercury temperature in separator, °F	500	
Mercury pressure in separator, psia	15	
	<u>Iron Section</u>	<u>Columbium Section</u>
Weight of wool, lb	4.06	8.32
Void volume, in. <sup>3</sup>	117	107
Average mercury velocity, ft/sec	$3.04 \times 10^{-3}$	$3.34 \times 10^{-3}$
Average residence time, sec	329	299
Estimated surface area, ft <sup>2</sup>	88.7	146.8

2. Testing

a. Cyclone-Type Vapor-Phase Separator

The cyclone-type, vapor-phase, corrosion-product separator described in Ref. 16 (Section IV,B,4) was installed in CTL-2, as shown in Figure 19.

The test plan called for the loop to be brought to operating conditions similar to those for a base-line test (Run 10 in Table 9) without a separator. The boiler outlet pressure was to be set at 265 psig, by means of the adjustable nozzle, and the loop was to be operated for 100 hours, or until the pressure reached 300 psig. Neither the nozzle, the mercury flow rates, nor any other controls were to be adjusted during the run. A constant boiler outlet pressure for 100 hours would indicate that the cyclone separator was effective in preventing corrosion-product buildup in the nozzle.

The loop operated for approximately 8 hours after the boiler outlet pressure was set at 265 psig, at which time the pressure reached 300 psig and the loop was shut down.

The fact that about the same amount of time was required to raise the nozzle inlet pressure from 265 to 300 psig with and without the cyclone separator (see Table 9) indicates that this device, as installed, was not effective in preventing corrosion-product deposition in the nozzle. The system operating conditions were comparable, if not exactly equal, throughout the test periods.

The separator was removed from the loop and sectioned. No deposit was found in the cyclone portion. It was postulated that such a deposit would be found if liquid droplets were thrown against the wall and dried during testing. There was evidence that a pool of liquid mercury remained in the collector pot and that some mercury refluxing occurred on the sides of the pot. The presence of liquid in the collector pot indicates that the experimental conditions did not provide a satisfactory test for the cyclone. The startup procedure also interfered with separator operation because of the requirement that the separator be full of liquid mercury, which then had to be boiled out after startup.

b. Hot Start of CTL-2

The startup technique used up to and including Run 11 involved filling the entire loop with liquid mercury. To provide a better test for future separators, a hot-start procedure was established that more closely simulates SNAP-8 system operation. This procedure consists of (1) evacuating the loop, (2) heating the boiler to approximately 1300°F, and (3) starting the mercury flow into the boiler without filling the loop, so that the nozzle will be exposed only to mercury vapor.

Run 12 consisted of the following steps:

- (1) CTL-2 was successfully hot-started.
- (2) The loop was allowed to settle out, and the boiler outlet pressure was set at 265 psig by adjusting the nozzle.
- (3) The loop operated for 9 hours until the boiler outlet pressure reached 300 psig, indicating that corrosion products had accumulated at the nozzle.
- (4) The loop was shut down by turning off the mercury pump and the heat to the boiler, and by valving off the mercury system so that mercury could not fill the boiler.

This test demonstrated that circulating hot mercury through the loop prior to startup was not the cause of corrosion-product buildup at the nozzle, and that the buildup on the nozzle (as indicated by increased boiler outlet pressure) offered a desirable method of evaluating the effectiveness of corrosion-product-separation devices (see Table 10).

c. Subsequent Operation of CTL-2

CTL-2 modifications were made to permit testing of the impingement type of corrosion-product separator. The loop was started without a separation device to establish a base-line condition for the loop. It was desired to know the loop-operation time necessary to achieve a buildup on the adjustable nozzle that would increase the boiler outlet pressure from 265 psig to 300 psig.

Superheated mercury vapor could not be obtained during the run, apparently because of poor thermal wetting of the Haynes 25 boiler tube. The loop was shut down, hot-outgassed for 83 hours, and restarted. Superheated mercury vapor was still not obtained, and the loop was allowed to operate at less than the rated condition in efforts to "run in" the boiler. After 64 hours of operation, a leak developed on the NaK side of the mercury boiler and the loop was shut down.

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TABLE 1  
ELEVATED-TEMPERATURE TENSILE TESTS, \* UN ELDED AND WELDED 9Cr-1Mo ALLOY STEEL

Test** Temp °F	Strength, psi x 10 <sup>3</sup>			Modulus of Elasticity psi x 10 <sup>6</sup>	Elongation % in 2 in.***	Reduction in Area, %
	Proportional Limit	Ultimate				
		0.1% Yield	0.2% Yield			
				Unwelded		
1250	11.6	18.7	19.5	35.1	11.6	82.8
1300	6.5	10.6	11.2	15.5	10.6	79.9
1325	5.6	9.0	10.0	13.4	11.0	75.5
				Welded		
1250	6.0	10.8	12.2	14.7	10.9	97.8
1300	5.9	8.5	9.3	13.6	8.2	97.8
1325	5.8	7.7	8.4	10.7	4.6	91.4

**\*|**

\* Average of two tests at each temperature.

※

Specimens were held at test temperature for 10 min prior to each test.

\*\*\*

\*\*\* All welded specimens failed in parent material.

\*  
\*  
\*  
\*

Failed outside of gage length.

TABLE 2

EVALUATION OF BRAZE ALLOYS (FOR JOINING OF 9Cr-1Mo ALLOY STEEL)

<u>Critical Temperature, °F</u>		<u>Lap-Joint Gap, in.</u>	<u>Ultimate Shear Strength psi</u>	<u>Alloy Flow on 9Cr-1Mo Specimen</u>	
<u>Liquidus</u>	<u>Solidus</u>				<u>Brazing Range</u>
General Electric Alloy No. J8100					
2075	1975	2125-2175	Surface contact	13,970	Good
			0.003	14,250	Good
			0.006	15,660	Good
General Electric Alloy No. J8400					
2100	2025	2150-2200	Surface contact	23,080	Poor
			0.001	21,100	Poor
			0.006	22,470	Poor
			0.010	21,930	Poor
AMS-4778 (Microbraz No. 130)					
1875	1800	1875-1950	Surface contact	30,203	Good
			0.001	20,700	Good
			0.003	14,140	Good
			0.006	14,330	Good
			0.010	13,890	Good

Table 2

TABLE 3

HYDROGEN CLEANUP AND BRAZING CYCLE\*  
WELDED AND BACK-BRAZED -1 MODEL CONDENSERS

Operation	Temp, °F	Time, hours:min	
		Incremental	Cumulative
Cleanup Cycle			
Heat	75 - 700	4:53	4:53
	700 - 1300	2:25	7:18
	1300 - 1400	1:00	8:18
	1400 - 1800	2:15	10:33
Hold	1800	1:00	11:33
Furnace-cool	1800 - 1325	6:21	17:54
Hold	1325	5:00	22:54
Furnace-cool	1325 - 1200	16:09	39:03
	1200 - 1000		
Purge hydrogen from retort with exothermic atmosphere and air-cool retort	700 - 75	2:10	41:13
Brazing Cycle			
Heat	75 - 700	3:50	3:50
	700 - 1300	2:32	6:22
	1300 - 1700	1:44	8:06
Stabilize	1700		
Heat	1700 - 1925	1:04	9:10
Hold	1925	0:05	9:15
Furnace-cool	1925 - 1750	0:35	9:50
	1750 - 1325	3:30	13:20
Hold	1325	5:00	18:20
Furnace-cool	1325 - 1200	20:10	38:30
	1200 - 700	17:50	56:20
Purge hydrogen from retort with exothermic atmosphere and air-cool retort	700 - 75	3:20	59:40

\* In -80°F dewpoint hydrogen atmosphere except as noted.

Table 3

TABLE 4

NOMINAL CHEMICAL COMPOSITION OF MATERIALS EXPOSED TO MERCURY-EROSION-TEST ENVIRONMENT

Material	Wt% of Major Elements												Cb+	
	C	Cr	Ni	Mo	Ti	Zr	Cb	Fe	W	Co	V	Cu	Ta	Al
TZM (Mo alloy)	0.02	-	-	Bal	0.5	0.08	-	-	-	-	-	-	-	-
Mo-0.5Ti	0.02	-	-	Bal	0.5	-	-	-	-	-	-	-	-	-
Cb-752	(40 ppm)*	-	-	-	-	2.5	Bal	-	10	-	-	-	-	-
17-4PH	0.07*	16.5	4.0	-	-	-	-	Bal	-	-	-	4.0	0.30	-
Lapelloy	0.30	11.8	-	2.8	-	-	-	Bal	-	-	0.25	-	-	-
AISI 347	0.08*	18	10.5	-	-	-	**	Bal	-	-	-	-	-	-
AISI 410	0.15*	12.5	-	-	-	-	-	Bal	-	-	-	-	-	-
PH15-7Mo	0.09*	15.0	7.0	2.5	-	-	-	Bal	-	-	-	-	-	1.0
18-4-1 tool steel	0.75	4.0	-	-	-	-	-	Bal	18	-	1.0	-	-	-
Dynacut tool steel	1.23	3.75	-	8.7	-	-	-	Bal	1.8	8.2	2.0	-	-	-
Stellite 6B	1.15	30	1.2	1.5	-	-	-	0.8	4.5	Bal	-	-	-	-

\* Maximum.

\*\* Ten times the carbon content.

Table 4

TABLE 5

## HEAT TREATMENT OF MERCURY-EROSION-TEST MATERIALS

Material	Heat Treatment	Room-Temperature Hardness	
		Rockwell B	Rockwell C
TZM (Mo alloy)	Stress-relieved, 2250°F for 1/2 hour, air-cooled	--	28
Mo-0.5Ti	Stress-relieved, 2100°F for 3/4 hour, air-cooled	--	29
Cb-752	Stress-relieved, 2200°F for 1 hour, air-cooled	84	--
17-4PH	900°F for 1 hour, air-cooled	--	44
Lapelloy	2000°F for 1 hour, oil-quenched 1200°F for 2 hours, air-cooled	--	34
AISI 347	1950°F, water-quenched	71	--
AISI 410	1550°F, furnace-cooled 50°F/hour to 1100°F, air-cooled from 1100°F	95	--
PH15-7Mo	1750°F for 10 min, air-cooled, -100°F for 8 hours, 950°F for 1 hour, air-cooled	--	48
18-4-1 tool steel	2250°F, oil-quenched, 1200°F for 1 hour, air-cooled, 1200°F for 1 hour, air-cooled	--	55
Dynacut tool steel	2200°F, oil-quenched, 1100°F for 2 hours, air-cooled, 1100°F for 2 hours, air-cooled	--	66
Dynacut tool steel	2200°F, oil-quenched, 1200°F for 2 hours, air-cooled, 1200°F for 2 hours, air-cooled	--	62
Dynacut tool steel	2200°F, oil quenched, 1200°F for 2 hours, air-cooled, 1200°F for 2 hours, air-cooled	--	54
Stellite 6B	2250°F, rapidly air-cooled, 1650°F for 4 hours, furnace-cooled to 700°F, air-cooled from 700°F	--	39

Table 5

TABLE 6

TEST CONDITIONS AND RESULTS  
MATERIAL SPECIMENS EXPOSED TO EROSIVE ENVIRONMENT

Material	Density lb/in. <sup>3</sup>	Original Weight g	Test Temp °F	Exposure Time hours	Weight Loss g	Calculated Volume Loss in. <sup>3</sup> x 10 <sup>5</sup>
TZM (Mo alloy)	0.369	9.8613	637	5	0.8023	479.0
Mo-0.5Ti	0.369	10.6385	653	5	0.7061	421.6
Mo-0.5Ti	0.369	9.7699	627	5	0.6707	400.1
Cb-752	0.326	7.1344	620	3.5	0.5867	396.4
17-4PH	0.281	7.9403	620	5	0.1448	113.5
Lapelloy	0.281	7.9118	620	5	0.1483	116.3
Lapelloy	0.281	7.9022	620	5	0.1403	110.0
Lapelloy	0.281	8.0322	620	4	0.1329	104.2
AISI 347	0.286	7.7620	628	5	0.1145	88.3
AISI 410	0.280	7.7548	629	5	0.0749	58.9
PH15-7Mo	0.271	7.5480	615	5	0.0393	31.25
18-4-1 tool steel	0.290	8.8753	620	5	0.0173	13.14
Dynacut tool steel	0.284	7.8966	620	5	0.0369	28.62
Dynacut tool steel	0.284	8.0063	620	5	0.0216	16.75
Dynacut tool steel	0.284	8.2336	639	14	0.0070	5.42
Dynacut tool steel	0.284	8.2336	655	5	0.0045	3.49
Stellite 6B	0.303	8.4745	636	15	0.0091	6.61
Stellite 6B	0.303	8.4770	625	15	0.0088	6.40
Stellite 6B	0.303	8.4745	650	5	0.0059	4.29
Stellite 6B	0.303	8.6966	620	5	0.0055	4.00
Stellite 6B	0.303	8.4770	637	5	0.0046	3.34

Table 6

TABLE 7TRANSDUCER PRESSURE AND TEMPERATURE DATA FROM RUN 11, COMPONENT TEST  
LOOP 2(CTL-2)

Time From Start of Test, hr	Transducer Number	Horizontal Leg			Corrected Pressure, psia	Transducer Number	Vertical Leg			Corrected Pressure, psia
		Temperature at Thermocouple Location, °F					Temperature at Thermocouple Location, °F			
		A	B	C			A	B	C	
	A <sub>1</sub>					B <sub>1</sub>				
0	Boiler Outlet	100	100	230	18	Boiler Outlet	110	110	340	17
1	"	185	210	575	137	"	180	285	390	140
3	"	205	225	560	170	"	No Reading			160
23	"	200	230	540	275	"	-	230	-	275
28	"	200	230	540	295	"	-	252	-	308
36	"	205	240	540	335	"	-	245	-	335
	A <sub>2</sub>					B <sub>2</sub>				
0	Nozzle Outlet	100	110	240	25	Nozzle Outlet	100	115	150	20
1	"	195	210	545	47	"	180	280	380	45
3	"	215	215	550	47	"	180	285	390	47
23	"	230	240	670	55	"	210	290	440	55
28	"	250	240	700	55	"	222	298	458	55
36	"	255	270	650	55	"	210	270	430	56
	A <sub>4</sub>									
0	Separator Inlet	100	120	220	20					
1	"	180	210	575	145					
3	"	200	225	550	275					
23	"	205	230	560	295					
28	"	205	230	565	305					
36	"	205	235	560	355					
	A <sub>5</sub>									
0	Boiler Inlet	100	110	340	51					
1	"	120	135	470	190					
3	"	130	140	475	220					
23	"	130	160	485	330					
28	"	135	165	490	390					
36	"	140	170	495	410					

Diagram illustrating the Vertical Leg setup. The diagram shows a vertical pipe with a cooling leg on the left side. Three thermocouples, labeled A, B, and C from bottom to top, are positioned along the right side of the vertical leg. A transducer is connected to the bottom of the vertical leg.

VERTICAL LEG

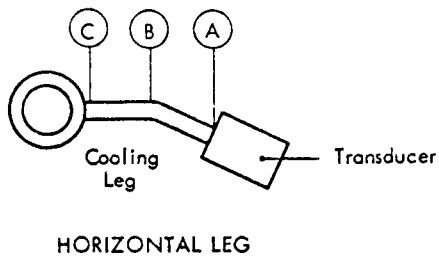
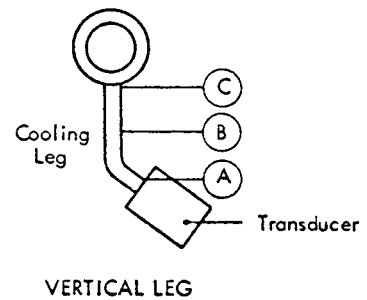
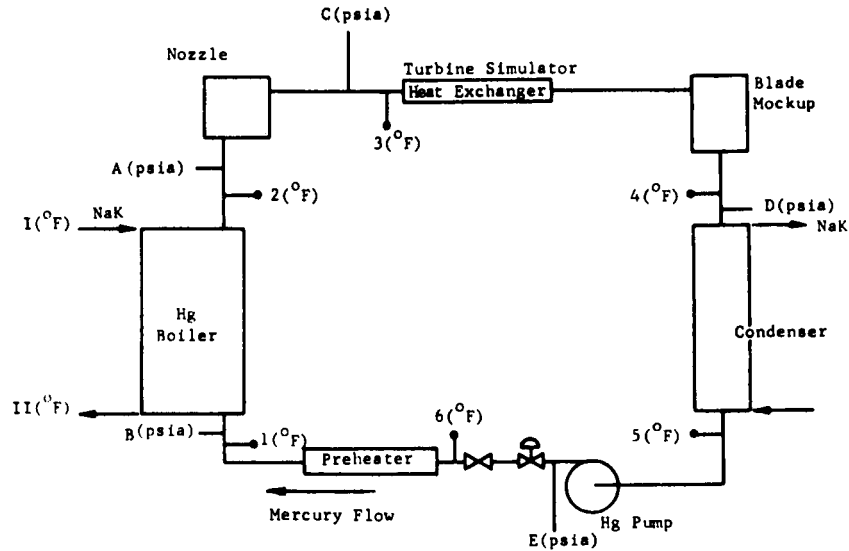


Table 7



TABLE 8

## TYPICAL OPERATING CONDITIONS OF CORROSION LOOP 3



Time From Start Of Hg Boiling Hrs	Hg Boiler						Condenser					Hg Flow #/hr	E psia	6 °F	Remarks
	1 °F	2 °F	A psia	B psia	I °F	II °F	3 °F	C psia	4 °F	D psia	5 °F				
3	430	1050	250	270	1300	1185	540	-	260	-	260	600	560	410	
14	440	1050	256	284	1280	1085	580	-	380	3.5	280	600	560	440	
38	470	1075	265	290	1325	1150	710	-	720	27.5	260	500	560	470	
59	440	1060	256	288	1310	1200	-	-	770	50	200	450	580	445	
70	Loop Shut Down Because Of High Condenser Inlet Pressure														Loop Repaired & Restarted
73	450	890	93	125	1270	1175	565	3	545	3	390	300	560	380	
97	600	890	95	125	1210	1125	590	6	570	4.5	375	300	560	400	
146	840	955	145	168	1300	1210	610	10	600	6.0	395	300	540	390	
188	800	950	140	175	1300	1200	650	12	600	6.5	385	300	532	400	
193	825	1105	130	165	1320	1190	950	10	605	6.6	410	300	540	405	Superheated Hg Obtained
195	760	1130	130	170	1300	1190	960	12	610	7.0	420	300	532	400	
202	810	1160	125	172	1300	1200	975	10	610	7.0	420	300	530	410	
266	750	1150	150	190	1320	1150	1000	13	640	10	470	410	530	430	
295	720	1175	165	210	1320	1150	1090	20	680	14	510	470	530	460	
319	680	1200	190	240	1320	1120	1120	24	680	15	520	590	546	460	
343	675	1205	270	300	1320	1130	1205	23	680	16	505	590	525	450	
348	Loop Shut Down Due To Mercury Flow Instability														Loop Restarted After a 10 Hr Shutdown
352	800	930	122	155	1310	1170	775	15	640	11	350	310	546	400	
354	770	1070	135	168	1310	1170	660	12	650	11	390	340	540	410	Superheated Hg Obtained
378	772	1100	133	165	1310	1150	935	14	655	12	420	340	540	418	
406	690	1060	180	220	1310	1120	720	20	670	14	510	500	530	460	
407	Loop Shut Down Due To Mercury Flow Instability														

Table 8

TABLE 9

## OPERATIONAL DATA FROM RUNS 10 and 11, CTL-2

Time from Start of Test hours	Flow Rate lb/hour	Boiler Pressure		Boiler		Temperature, °F			
		psig		Coil 2/3	Nozzle	Vapor-Phase Cyclone Separator		Collection Pot	
		Inlet	Outlet	Down	Outlet	Inlet	Outlet	Inlet	Outlet
		ΔP							
Run 10 (Without Cyclone Separator)									
0-40	During the first 40 hours, flow-temperature and choke-nozzle adjustments were made, and the last 11 hours were used for observation of boiler-outlet-pressure increase with time.								
41	1000	330	270	60	1140	1210	1130	-	-
45	1000	350	275	75	1140	1210	1135	-	-
47	1000	360	280	80	1140	1210	1135	-	-
50	1000	370	300	70	1140	1210	1135	-	-
51	1000	375	305	70	1140	1210	1135	-	-
Run 11 (With Cyclone Separator)									
1	1200	68	40	28	460	520	190	500	100
3	1100	190	125	65	670	795	290	690	205
6	1000	230	135	95	830	970	660	850	395
10	1000	260	175	85	1080	1160	940	1055	445
14	1000	275	200	75	1090	1168	1050	1090	750
18	1000	295	225	70	1085	1170	1100	1090	1010
24	1000	330	265	65	1095	1190	1130	1090	1200

(cont.)

TABLE 9 (cont.)

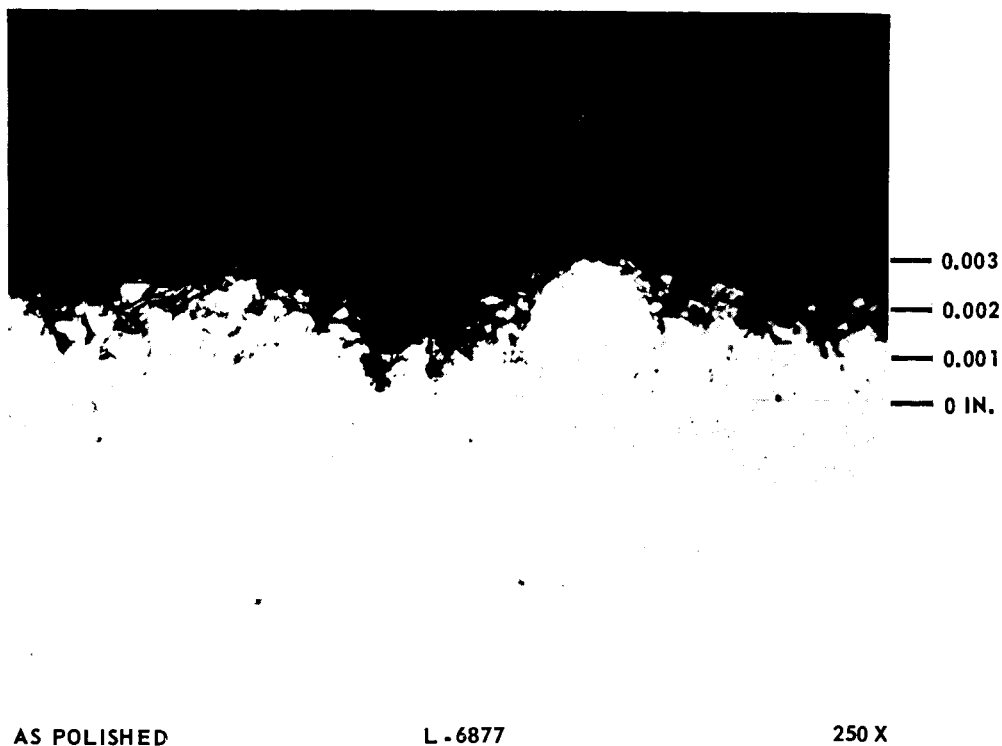
[illegible]

TABLE 10

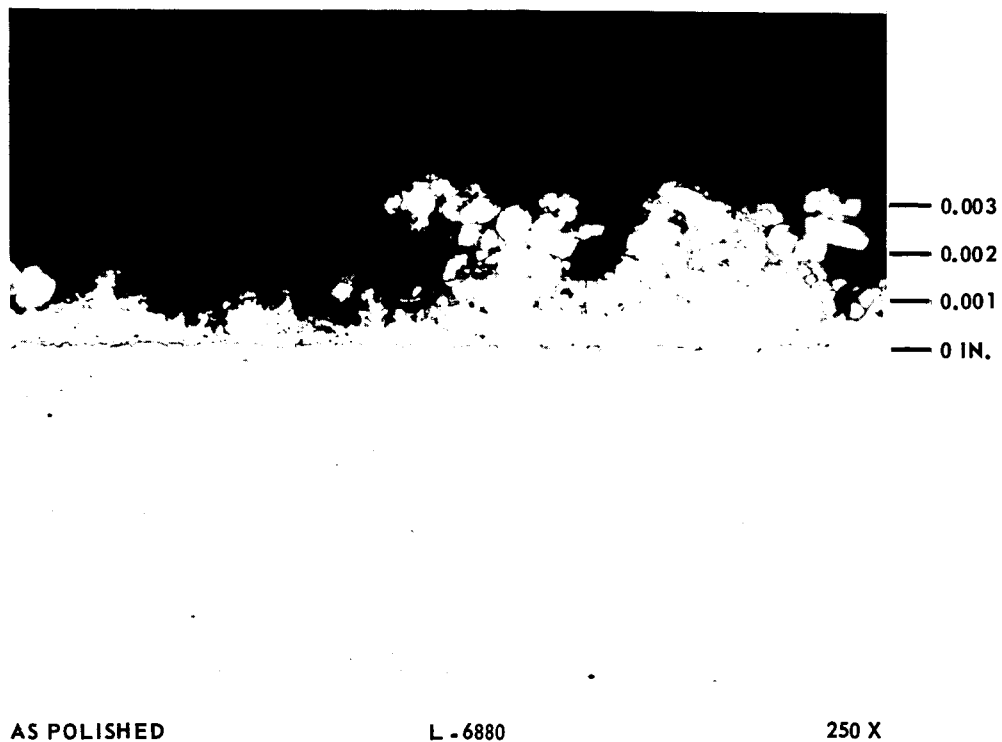
HOT-START DATA FROM RUN 12, CTL-2

Time from Start of Test hours	Flow Rate lb/hour	Temperature, °F									
		Boiler Pressure		Boiler		Nozzle		Preheater		Mercury Pump	
		Inlet	Outlet	psig	ΔP	Coil 2/3 Down	Outlet	Inlet	Outlet	Inlet	Outlet
0	1200	270	190	80	80	1200	1250	500	800	200	385
1	900	250	200	50	50	1150	1200	530	820	200	390
2	900	275	260	15	15	1220	1290	530	830	210	
3	1000	310	250	60	60	1180	1270	540	830		
4	1000	330	270	60	60	1200	1250		820		
5	1100	340	275	65	65		1250				
6	1100	345	280	65	65		1250				
7	1000	350	285	65	65		1240				
8	1000	350	290	60	60		1240				
9	1000	350	300	50	50		1240				
9.5	1000	350	300	50	50		1240				

Table 10

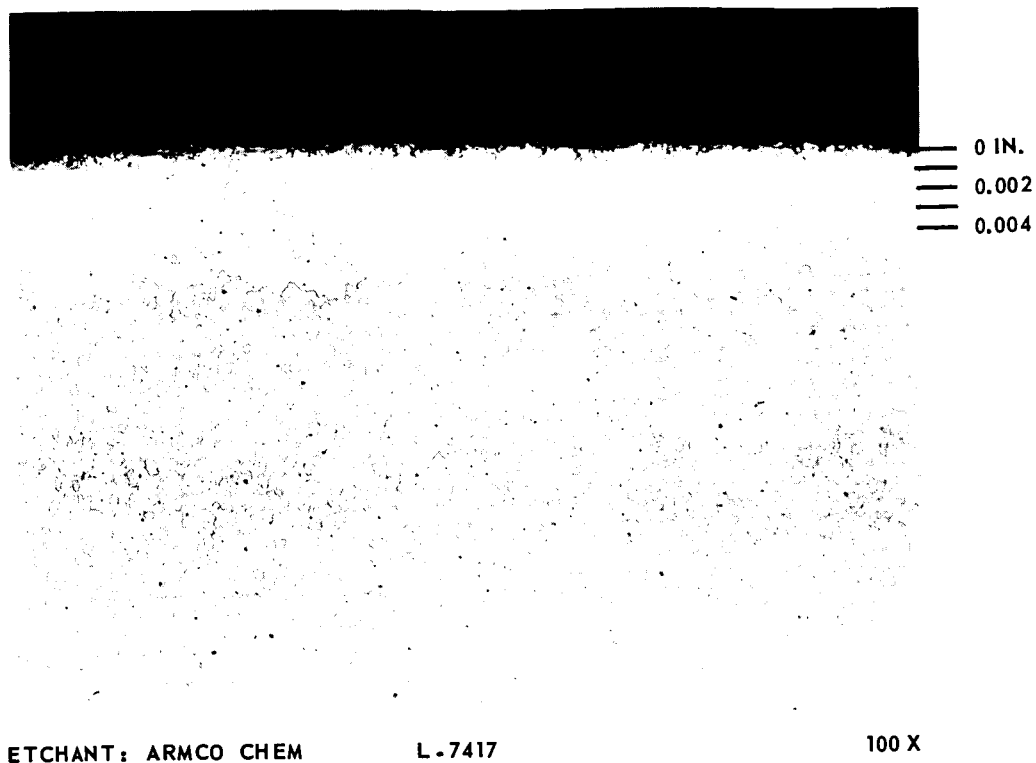


(a) Interior Surface of Tube

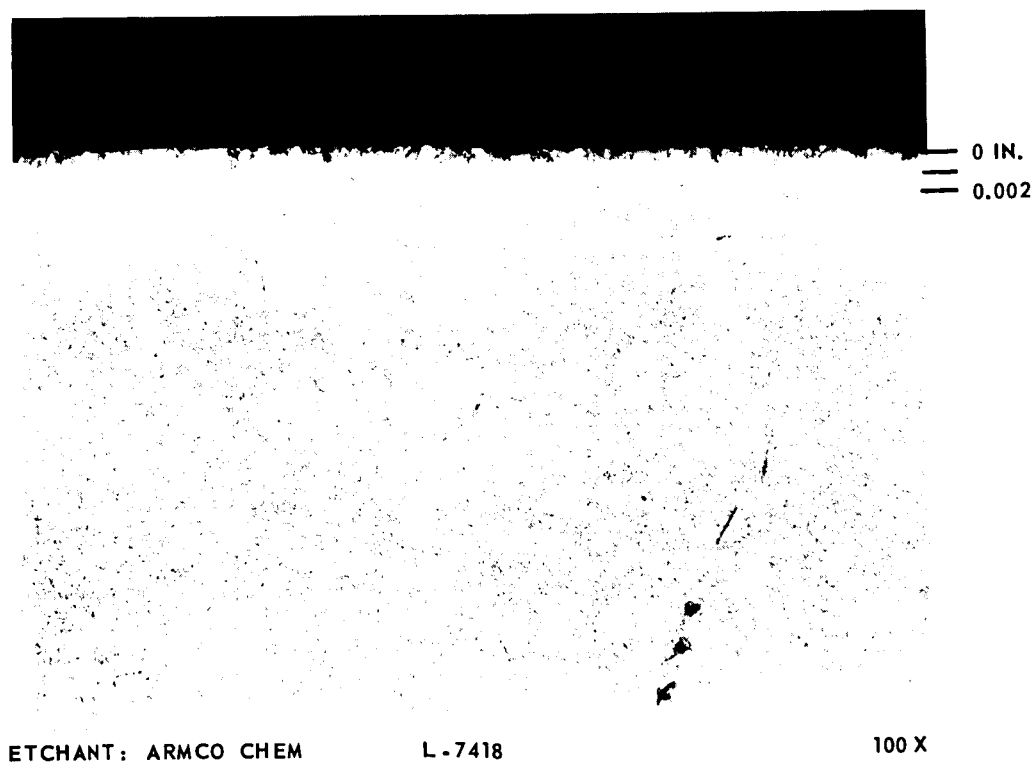


(b) Surface of Twisted Ribbon in Tube

Mass-Transfer Deposit in Mercury-  
Containment Tube (9Cr-1Mo Steel) of TS-2B Boiler After 269 Hours  
of Operation (Approximately 45 ft from Mercury Inlet and 90% Vapor Quality)



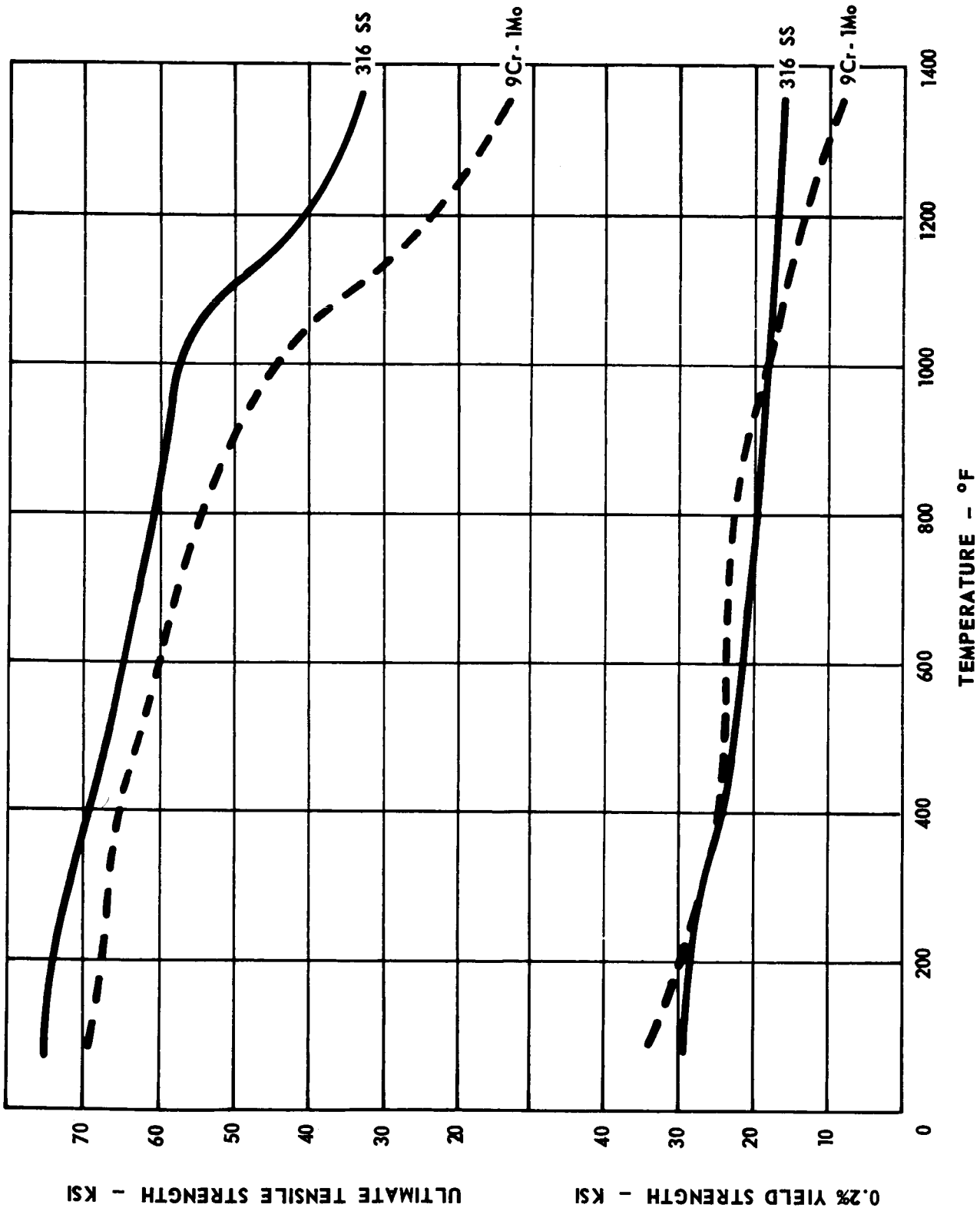
(a) Segment 12, at NaK Inlet



(b) Segment 8, Approximately 30 ft from NaK Inlet

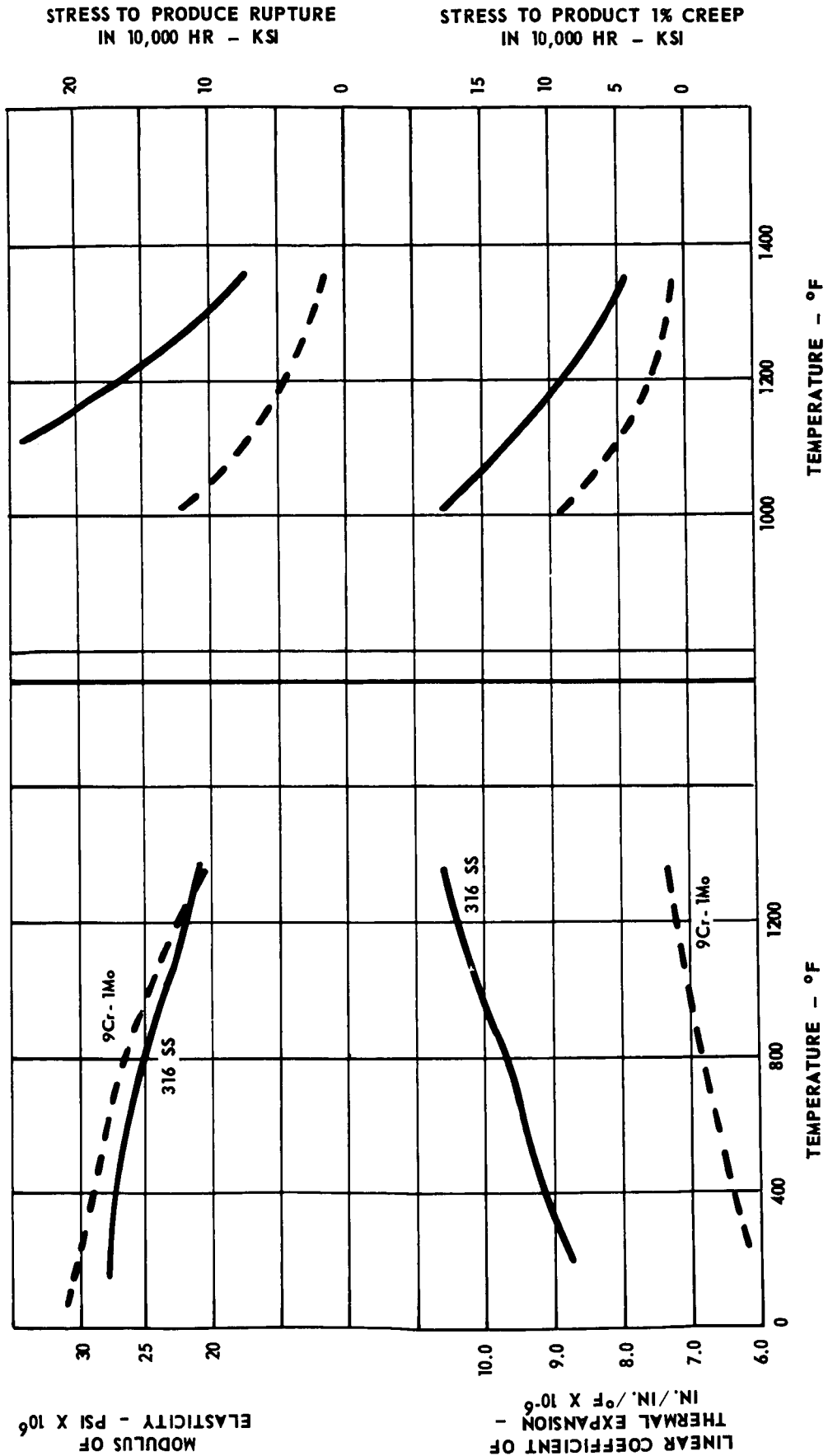
Exterior Surface (Exposed to 1300°F NaK) of Two Locations on  
Mercury-Containment Tube (9Cr-1Mo Steel) of TS-2B Boiler, Showing Surface  
Decarburization After 269 Hours of Operation

Figure 2



Yield Strength and Ultimate Tensile Strength of Wrought 316 SS (Annealed) and 9Cr-1Mo Steel (Normalized at 1700°F and Tempered at 1350°F) to 1350°F

Figure 3



Various Mechanical and Physical Properties of Wrought 316 SS (Annealed) and 9Cr-1Mo Steel (Normalized at 1700°F and Tempered at 1350°F to 1350°F)

Figure 4



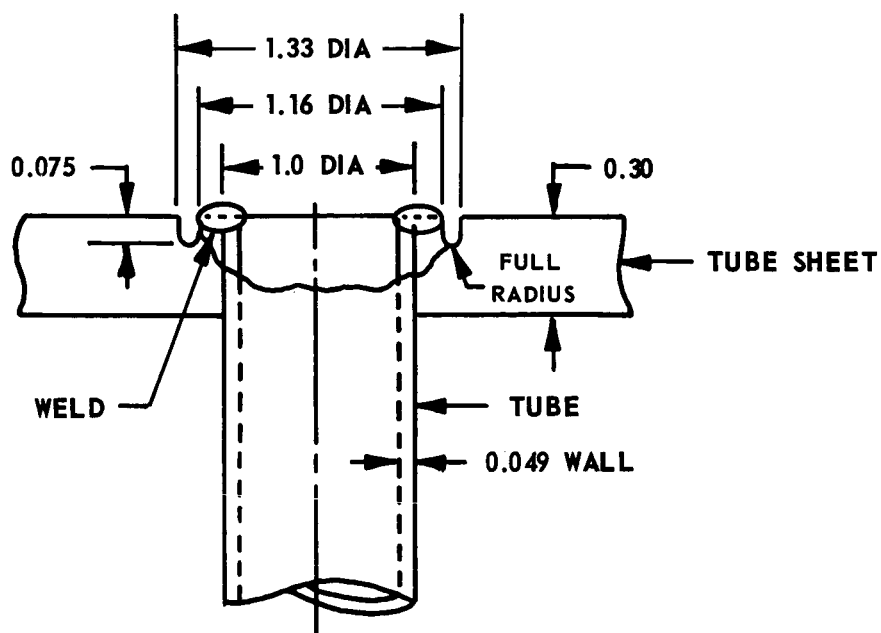


Fig. 5. Design of Joint Between Turbine-Simulator 9Cr-1Mo Steel Tube and Tube Sheet

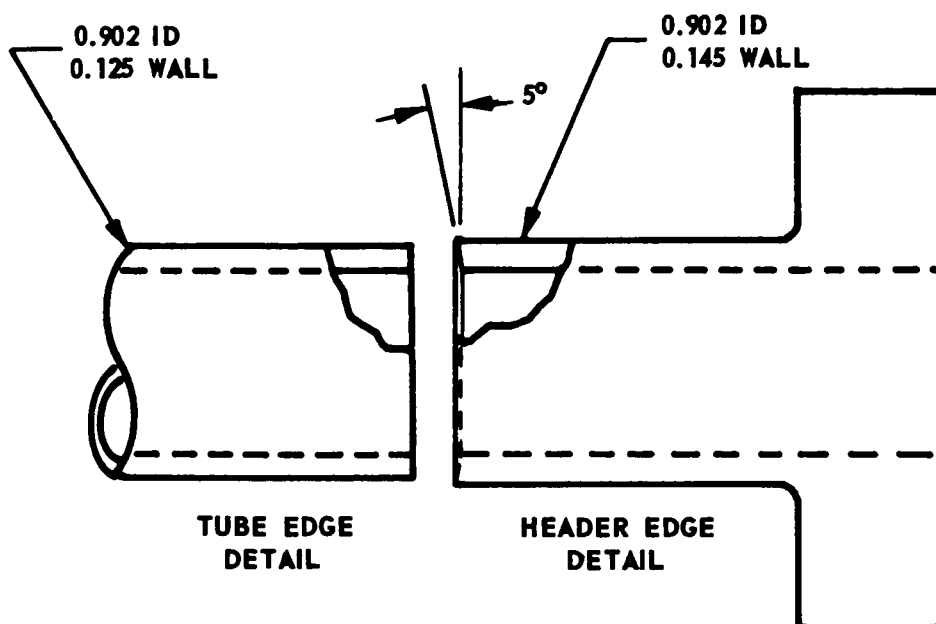
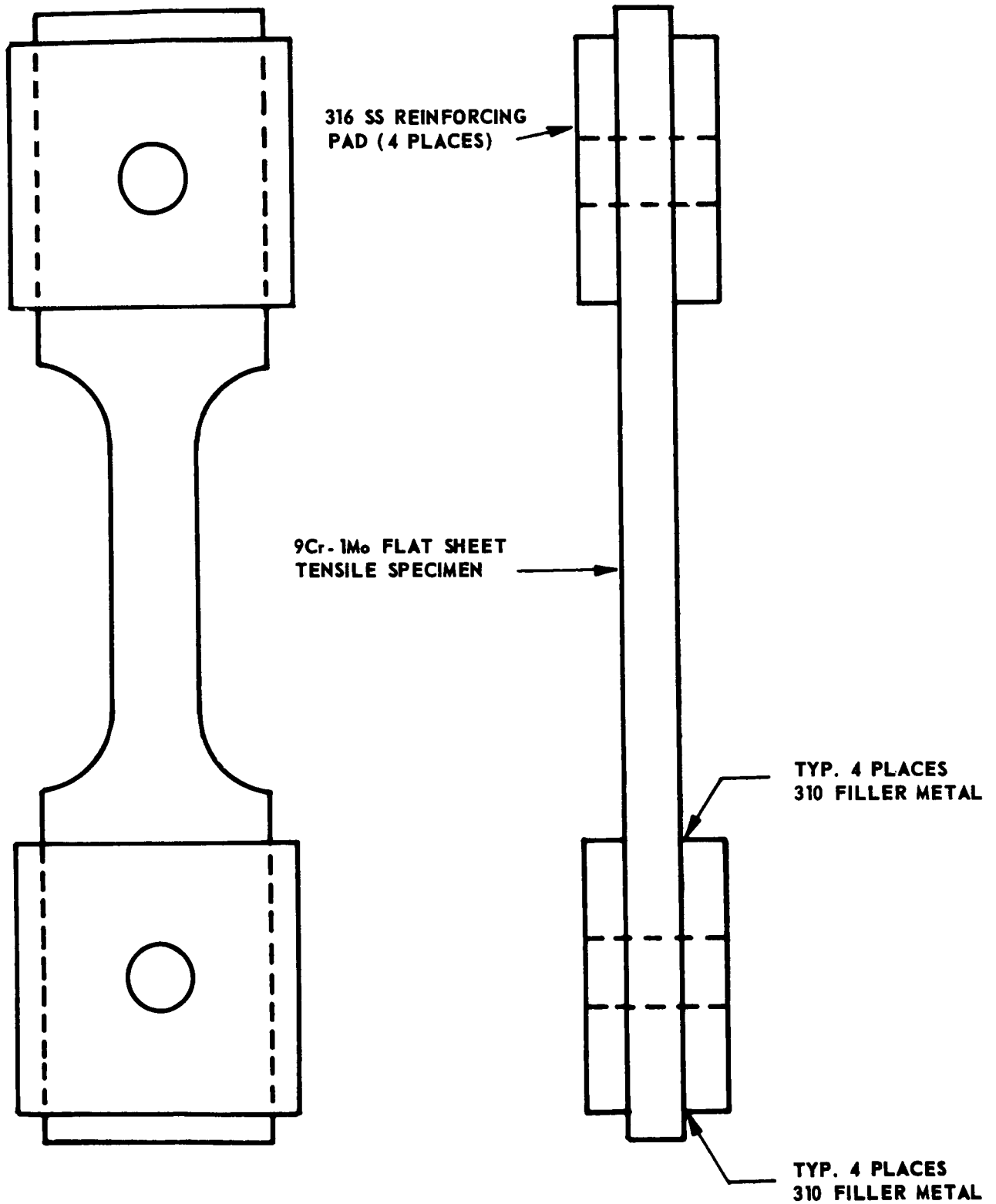
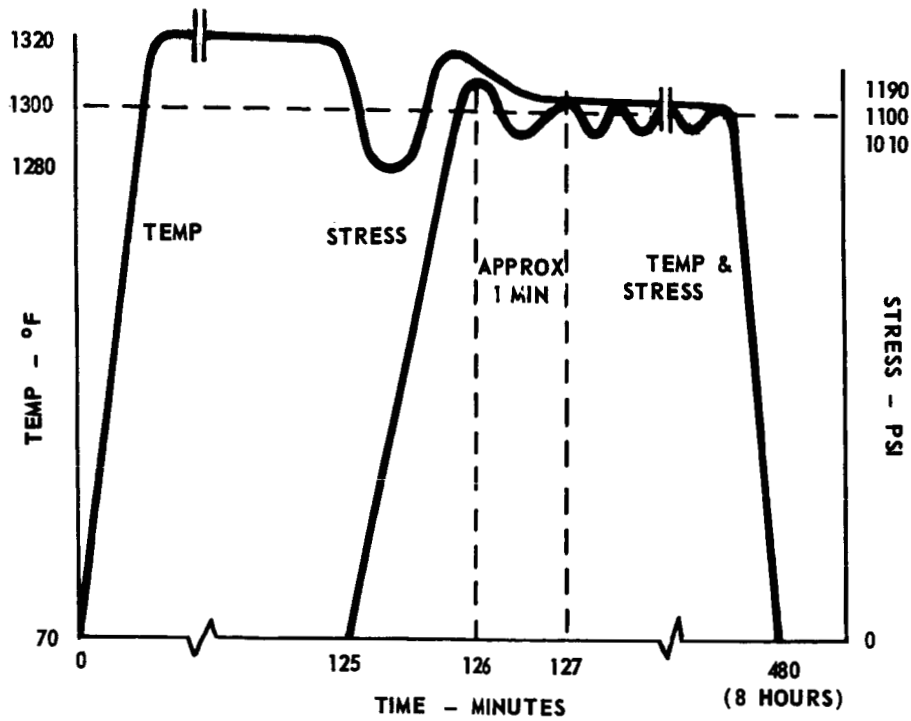


Fig. 6. Tube-to-Header Joint Design for Internal Tube Weld on -1 Model Boiler

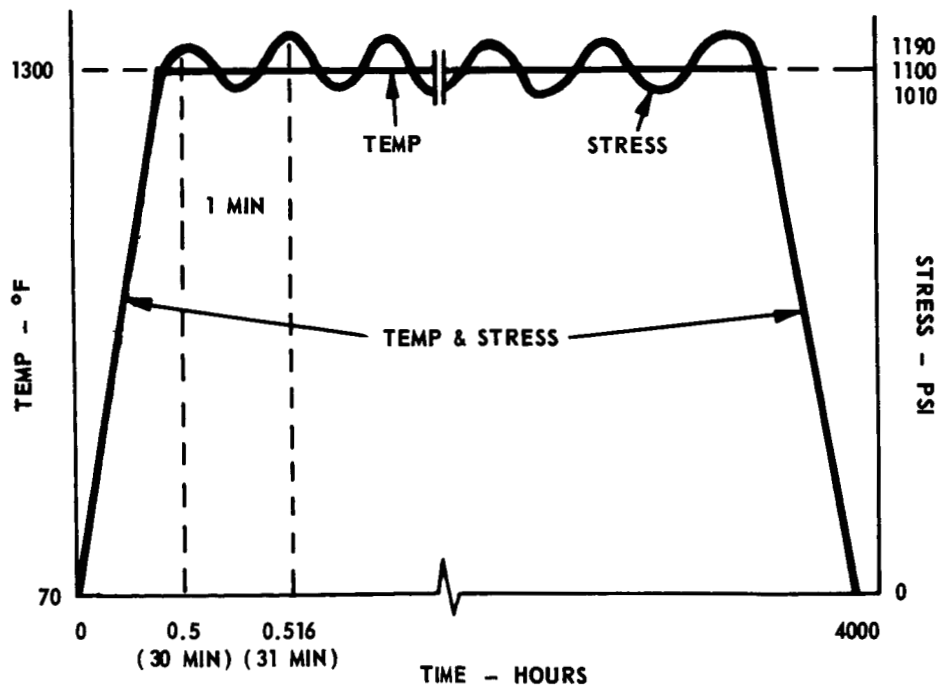


Flat Tensile-Test Specimen of 9Cr-1Mo Steel  
with Grip Area Reinforced by 316 SS Pads

Figure 7

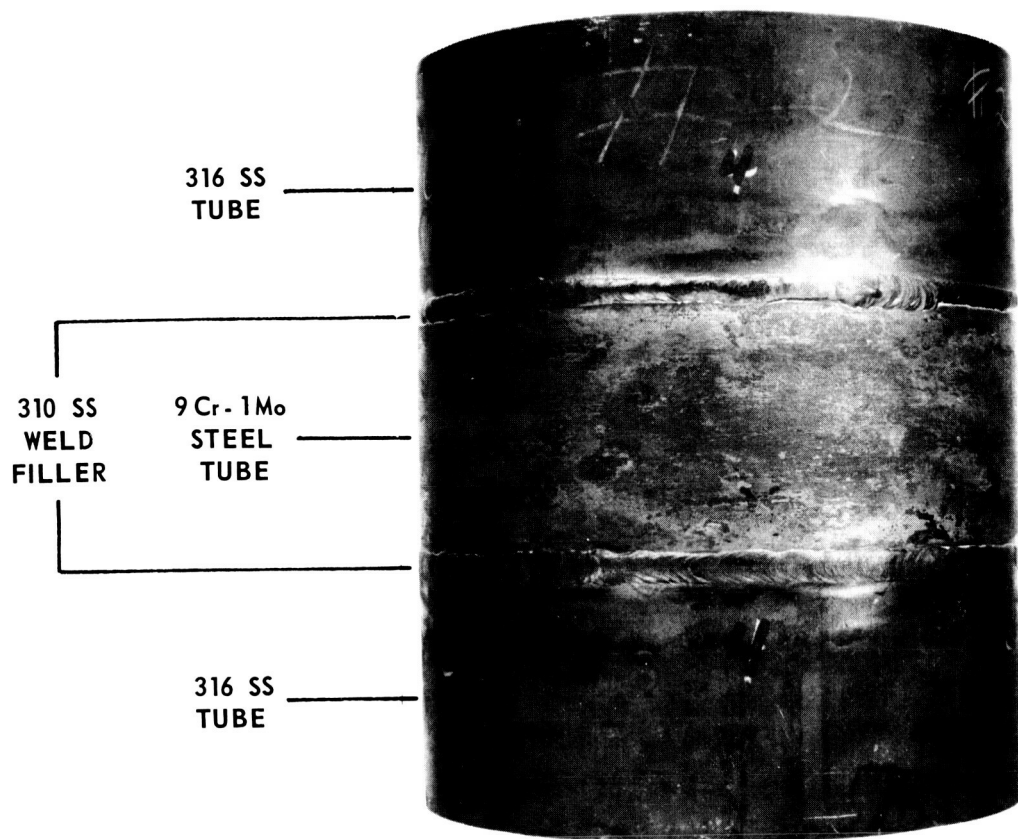


(a) Phase I - SNAP-8 System, Startup-Cycle Simulation  
(25 Cycles Required)



(b) Phase II - SNAP-8 System, Continuous-Operation Simulation

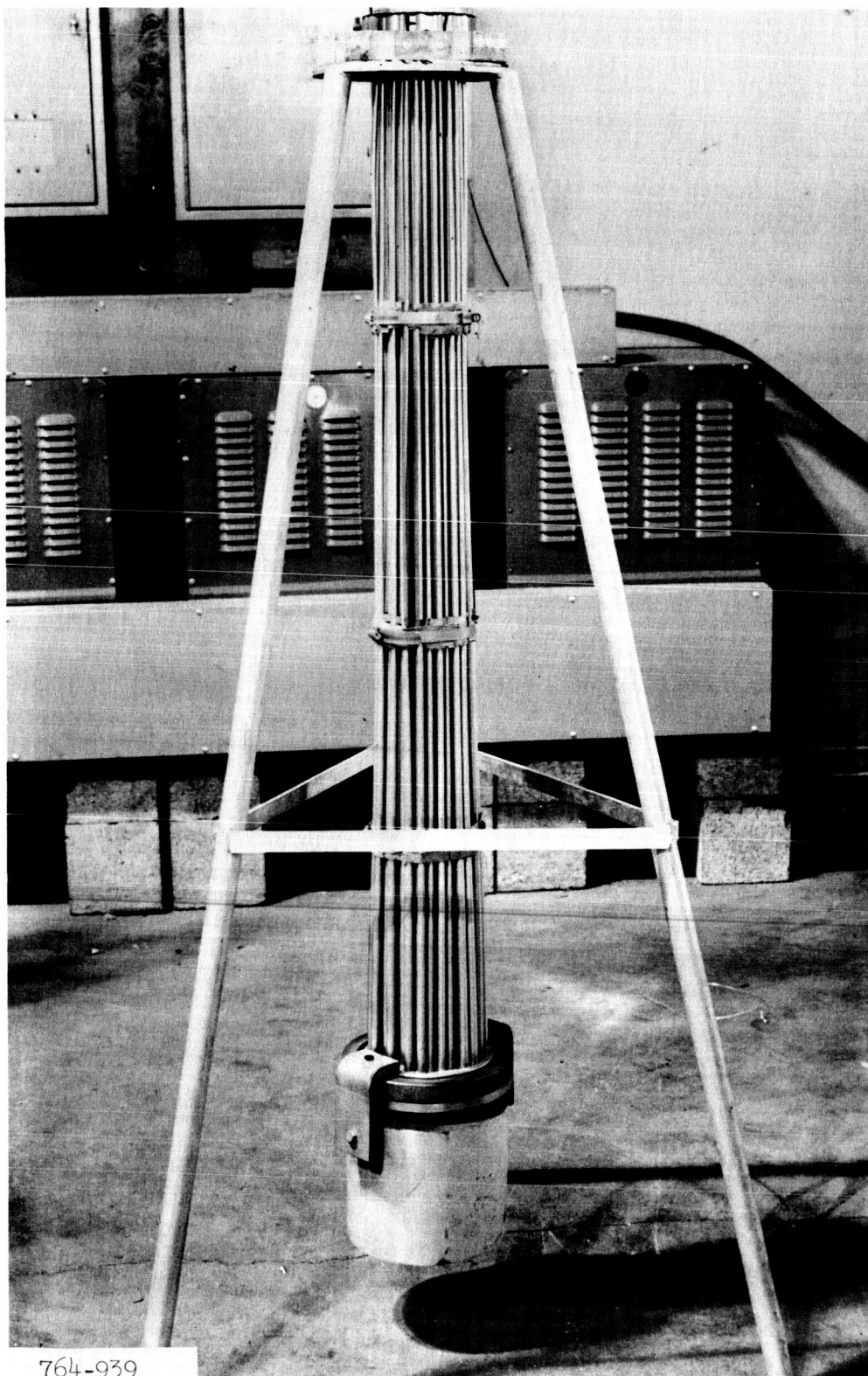
Temperature and Pressure Cycling Program for 9Cr-1Mo/316 SS  
(310 SS Weld-Filler Material) Transition-Joint Evaluation



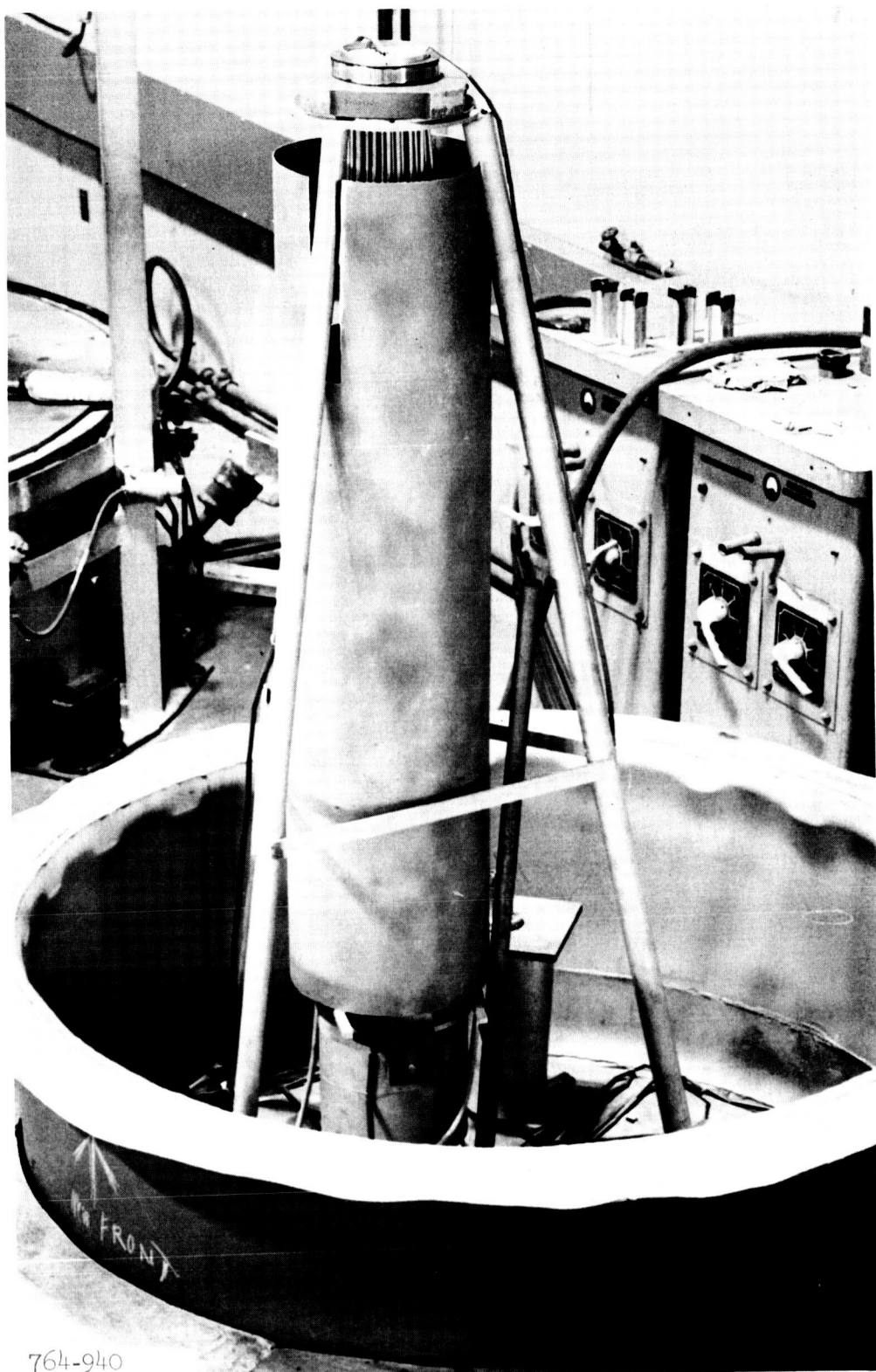
L-7066

2/3 X

As-Fabricated Specimen of 9Cr-1Mo/316 SS  
for Transition-Joint Evaluation

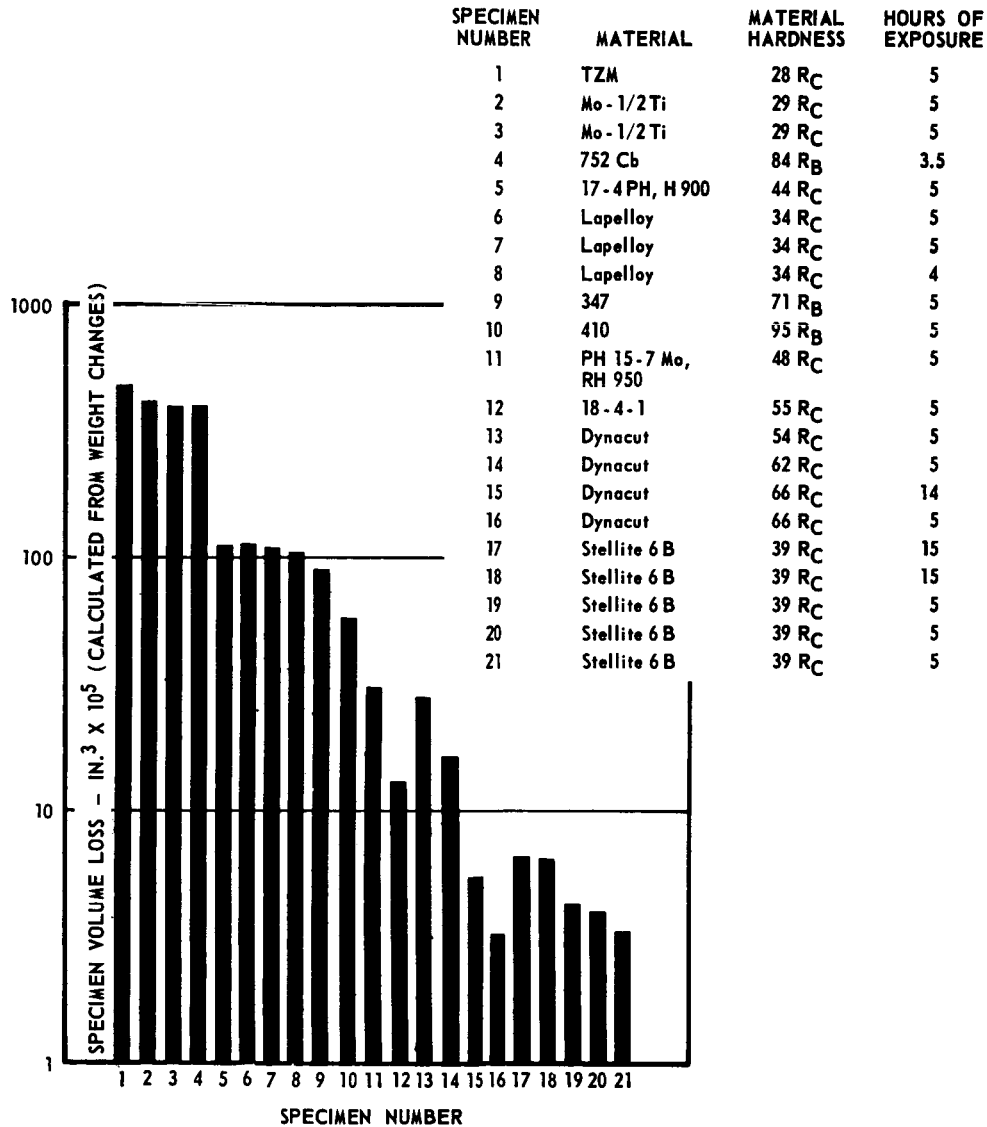


-1 Model Condenser-Tube-Bundle Assembly Suspended from  
Small Header in Brazing Fixture

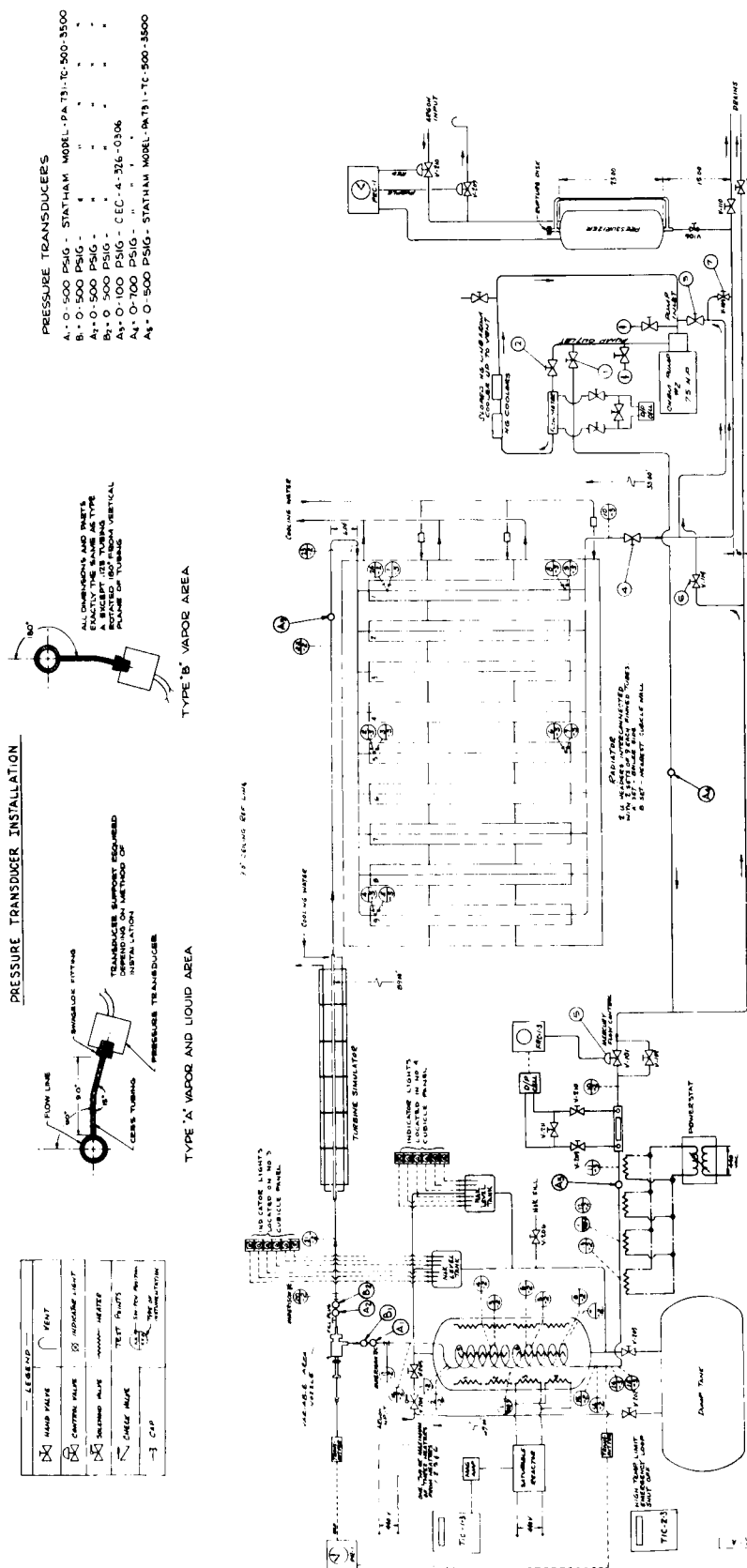


-1 Model Condenser-Tube-Bundle Assembly Suspended in  
Brazing Fixture, with Heat-Radiation Shield in Position

Figure 11



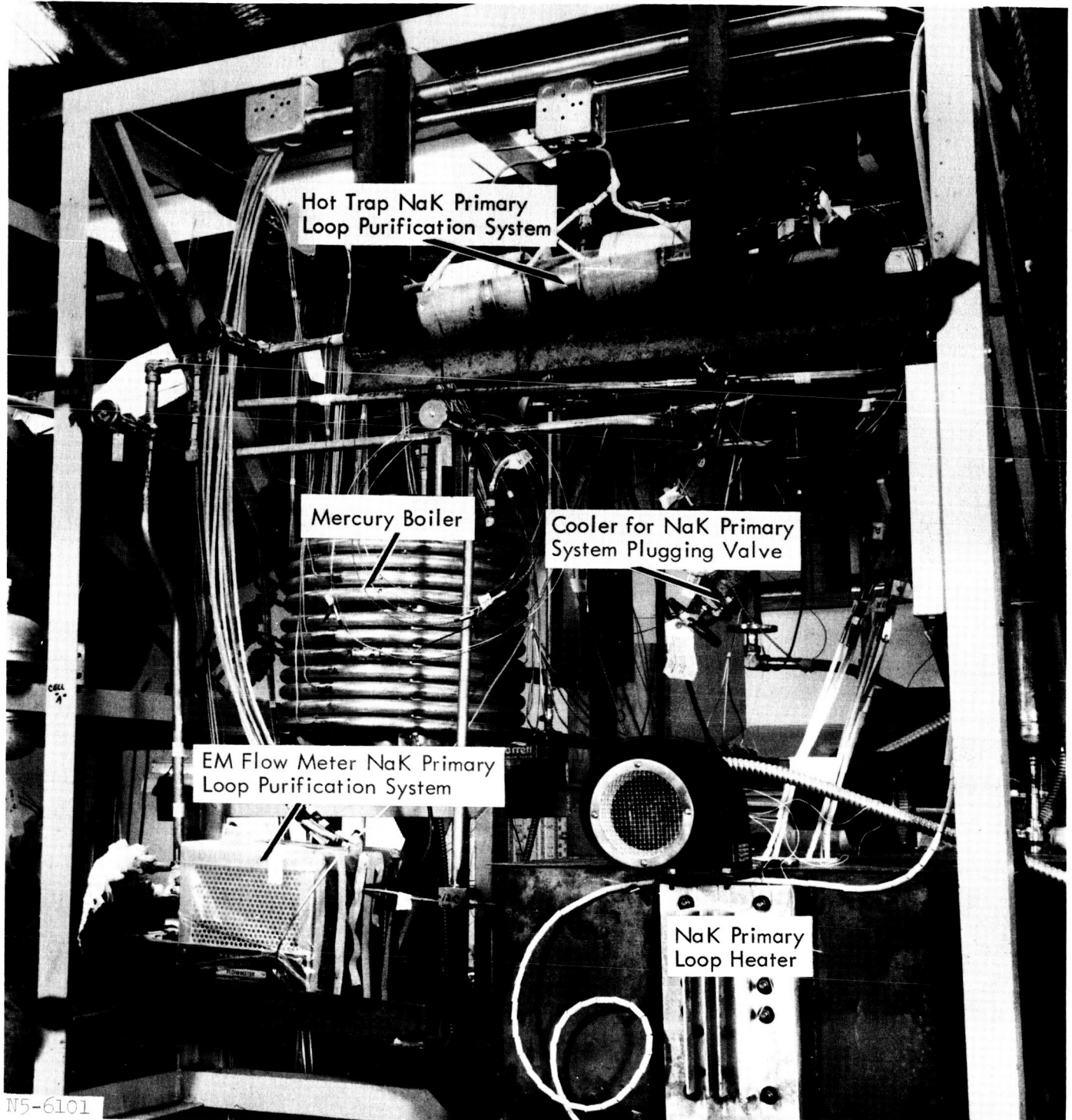
Volume Loss, Accelerated-Mercury-Erosion Test Specimens



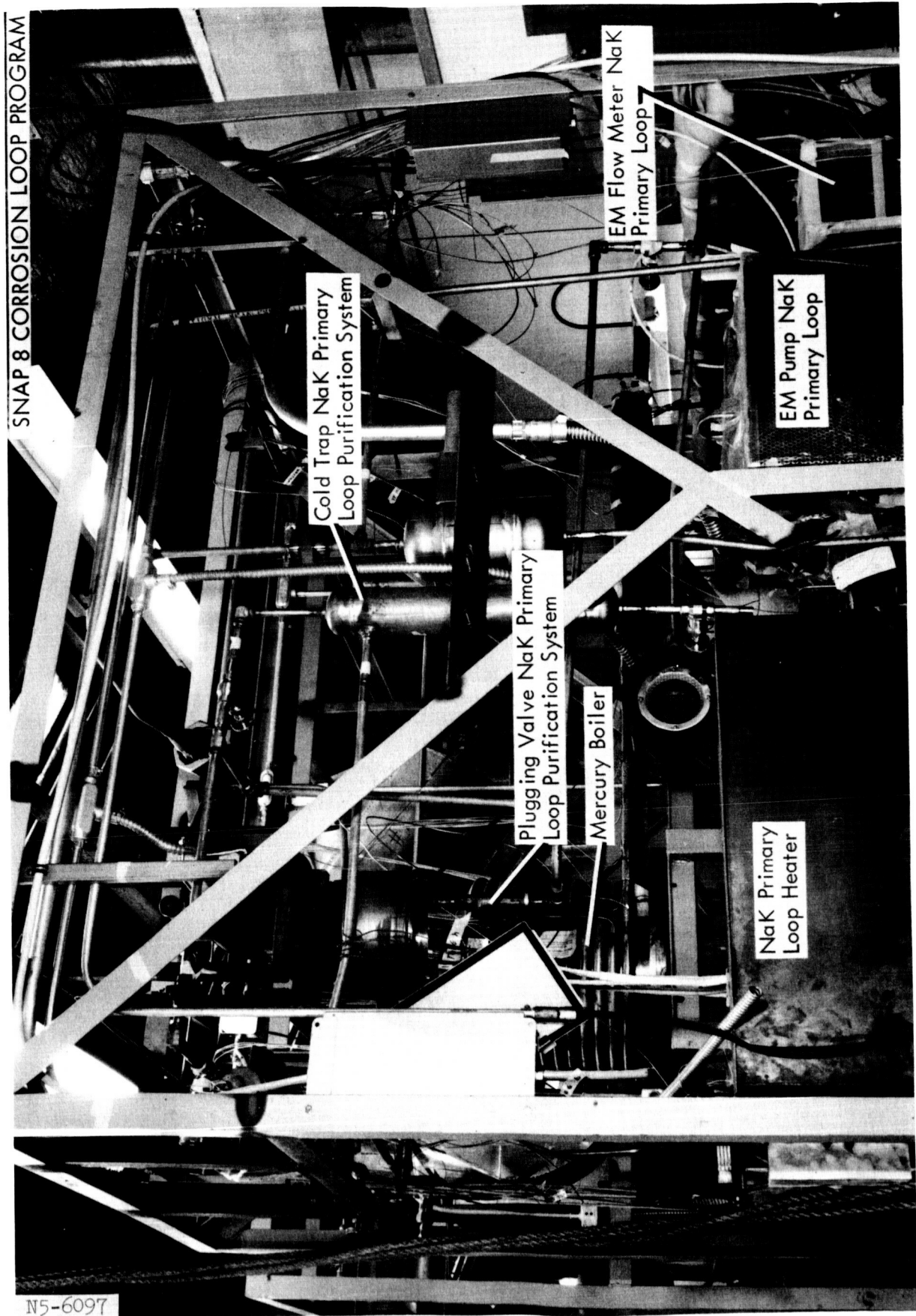
### Schematic Diagram of Component Test Loop 2, Showing Location of Pressure Transducers

Figure 13



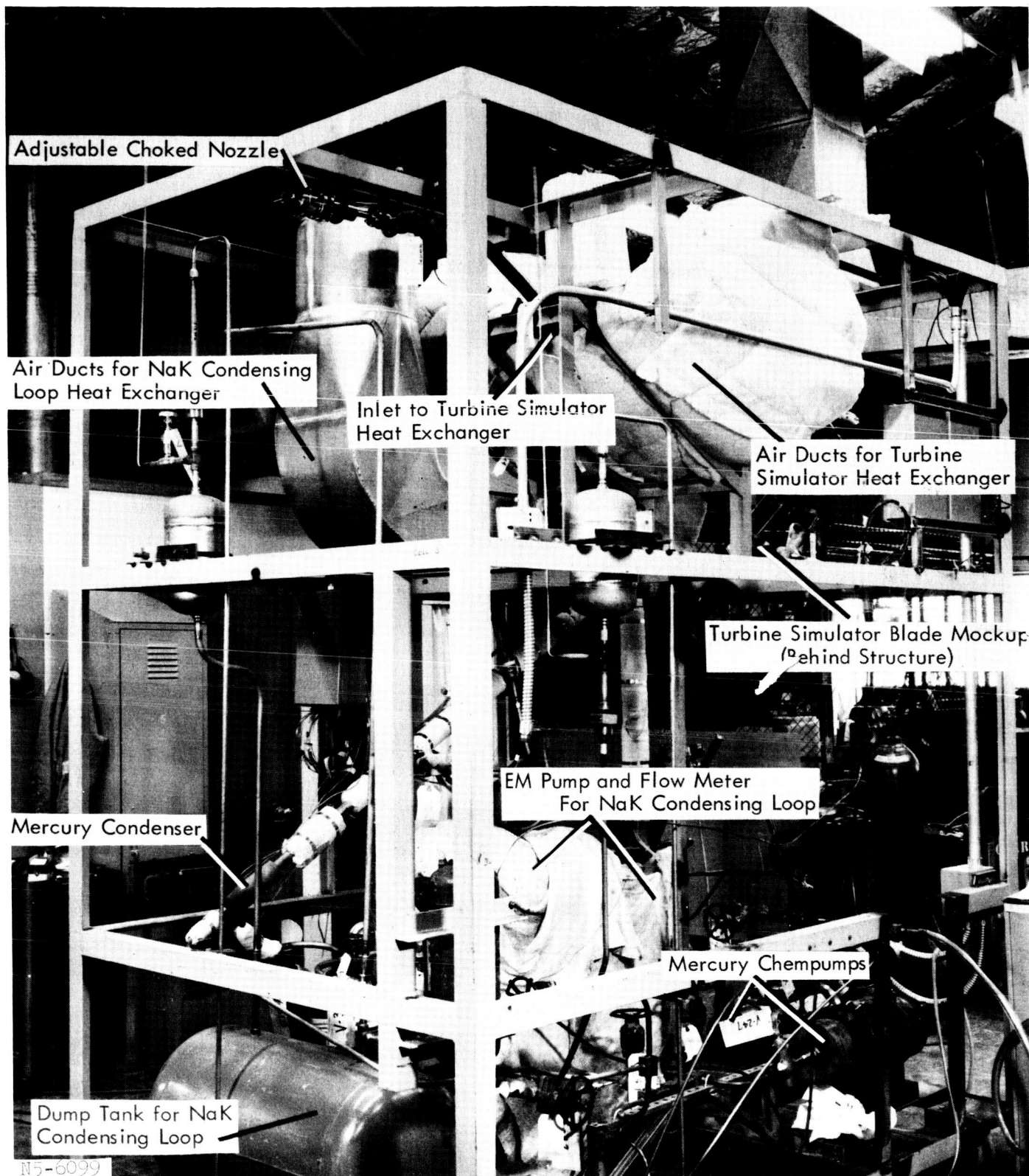


Corrosion Loop Assembly, Cell A (Front View)



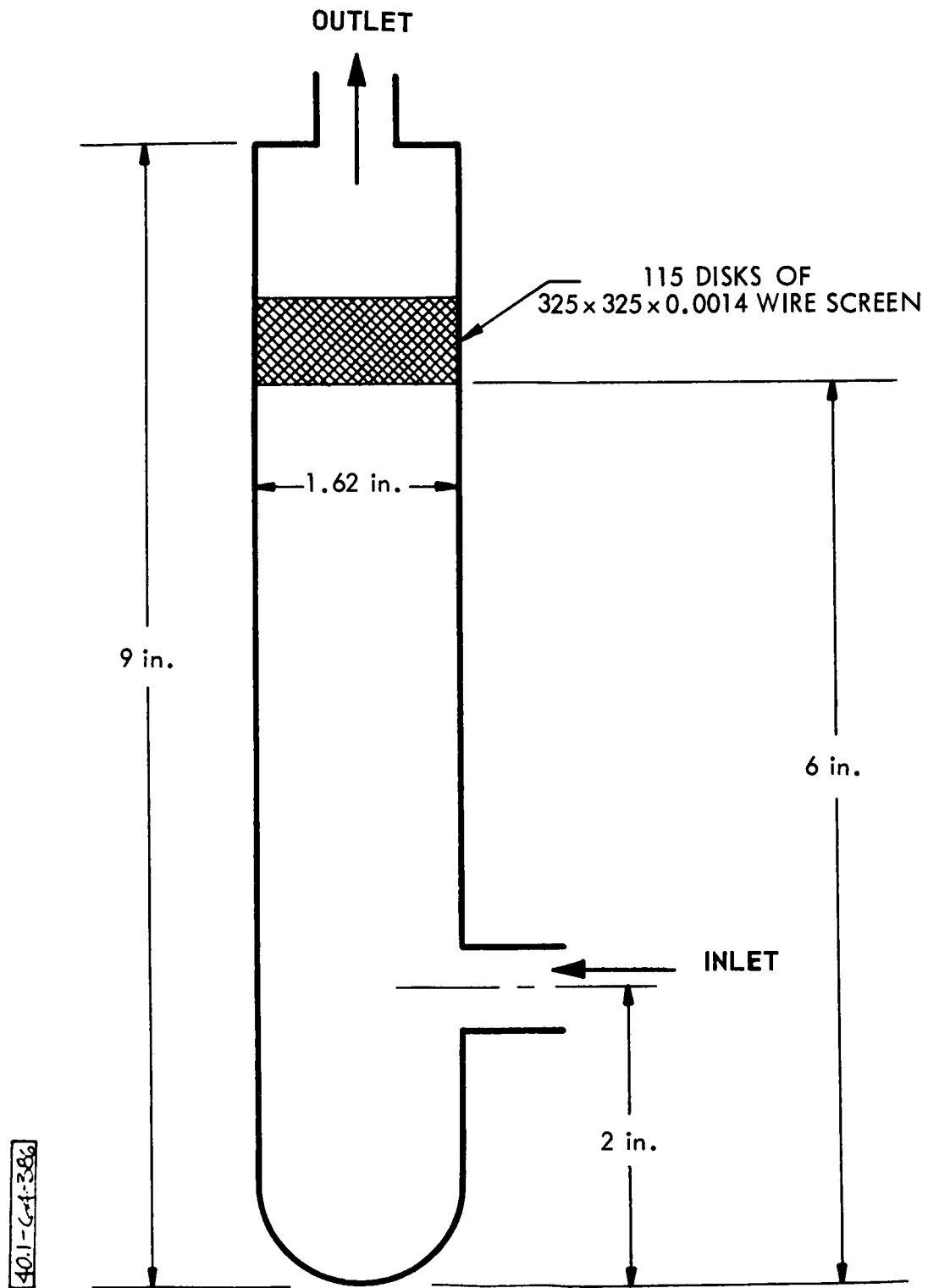
Corrosion Loop Assembly, Cell A (Side View)

Figure 15



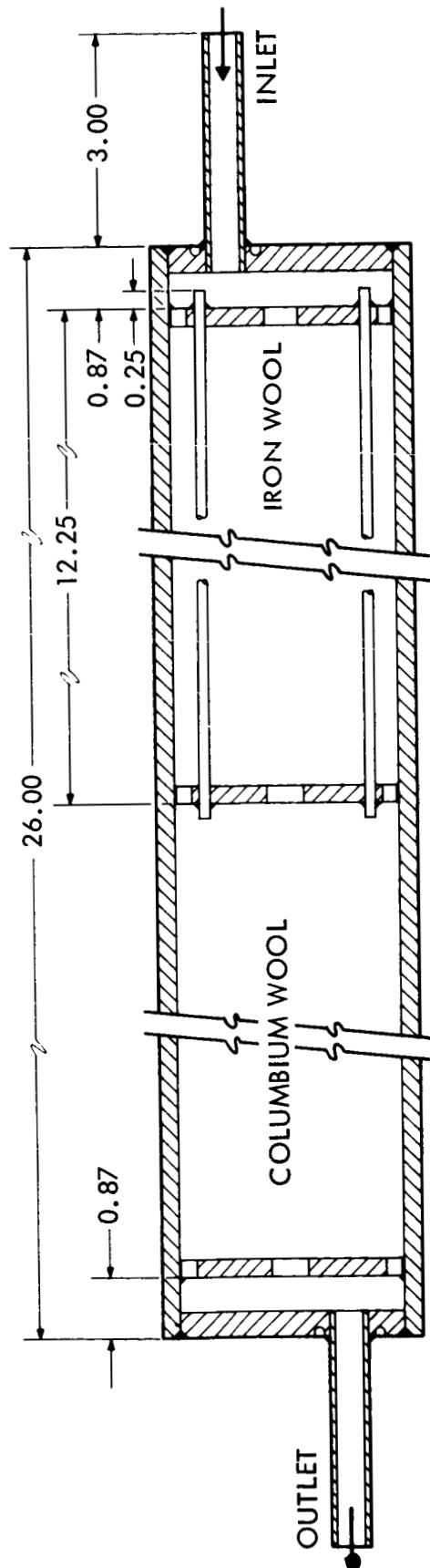
Corrosion Loop Assembly, Cell B

Figure 16



Impingement Separator

Figure 17



Liquid-Phase Mercury-Corrosion-Product Separator

Figure 18

40.1-64-1326

40.1-64-375

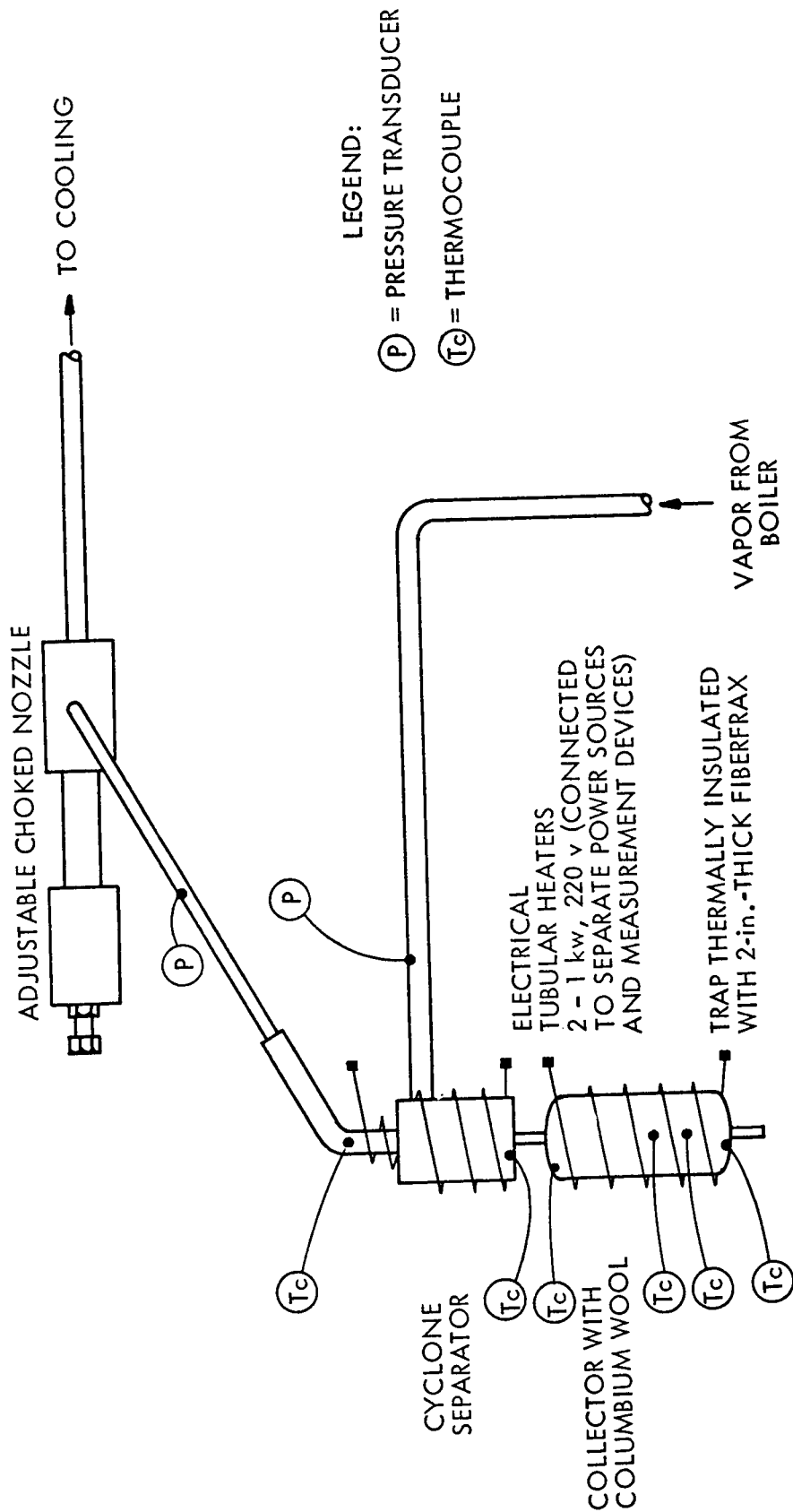


Figure 19

Mercury-Vapor Corrosion-Product Cyclone Separator, as Installed in CTL-2 for Run 11

APPENDIX

COLUMBIUM TUBING CLAD WITH STAINLESS STEEL  
(Engineering Data Sheet 390-64-0188B)

SCOPE: This engineering data sheet covers wrought columbium tubing clad with Type 316 stainless steel for use in the SNAP-8 Materials Development Program.

SIZE: As specified on the purchase order.

MATERIAL: Columbium tubing to be manufactured from electron-beam-melted ingot.

Composition limits for finished tubing:

Liner: Columbium of the following composition:

Carbon	200 ppm, max	Zirconium	500 ppm, max
Nitrogen	300 ppm, max	Iron	500 ppm, max
Oxygen	500 ppm, max	Tantalum	1000 ppm, max
Hydrogen	30 ppm, max	Titanium	500 ppm, max
Columbium	99.5%, min (by difference)		

Cladding material: Type 316 seamless stainless steel per ASTM A-269-58T.

TOLERANCES: Inside diameter,  $\pm 0.005$  in.  
Total wall thickness,  $\pm 10\%$   
Columbium wall thickness,  $\pm 10\%$   
Type 316 SS wall thickness,  $\pm 10\%$

TEMPER: The finished tubing shall be heat-treated so that a coil of 8-in. outside diameter can be made from the tubing without cracking or splitting the tubing materials. Carbide precipitation in the 316 stainless cladding is acceptable.

METHOD OF MANUFACTURE: The tubing is to be manufactured by co-extrusion. A metallurgical bond between the columbium and the stainless steel cladding is desired.

TEST METHODS: Formability: The criterion for a satisfactory metallurgical bond between the columbium and the stainless steel shall be the capability of passing the following test:

1. Bend a representative length (approx 12 in.) of finished tubing into an arc of 8 in. diameter.

2. Heat the bent section of tubing to 1400°F, hold for 1 hour, and cool to room temperature. Heating and cooling rates shall be 200°F per hour (minimum).
3. Microexamination at 500X of three random samples of a cross section of the tubing shall show no separation of the cladding from the columbium inner lining.

Ultrasonic inspection test: Each tube shall be ultrasonically inspected utilizing a method mutually acceptable to Aerojet-General and the vendor. The sum of all individual unbonded areas shall not exceed 5% of the total surface area of a tube length.

Hydrostatic test: Each tube shall be tested to a hydrostatic pressure sufficient to produce a fiber stress of 6000 psi in the total wall thickness.

**QUALITY AND  
FINISH:**

The finished tubes shall be visibly free from oxide, grease, oil, residual lubricants, and other extraneous materials.

The finished tubing shall be free of cracks, seams, slivers, blisters, burrs, and other injurious imperfections.

**REPORT  
REQUIRED:**

Chemical analyses of the columbium and stainless steel shall be supplied. Results of the formability test, including copies of the photomicrographs of the three sections examined for cladding separation, shall be supplied.



NASA CONTRACTOR REPORT

SNAP-8 MATERIALS REPORT FOR JANUARY-JUNE 1964

VOL. II - DEVELOPMENT OF COMPONENT MATERIALS

Aerojet-General Corporation

ABSTRACT

Investigations of potential SNAP-8 materials were conducted, in areas where the state of the art was insufficiently developed, (1) to permit reliable material selection, and (2) to assist in the design, development, fabrication, and testing of SNAP-8 components.

A mercury forced-convection corrosion-loop investigation was continued to evaluate the corrosion resistance of 9Cr-1Mo alloy steel for 10,000-hour life. The mercury-corrosion resistance of backup mercury-containment materials was also evaluated.

Data were developed to substantiate the selection of 9Cr-1Mo steel as a mercury-containment material and to facilitate an evaluation of potential problems related to its use.

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